VAMAS

VAMAS TECHNICAL WORKING AREAS

ANNUAL REPORTS

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May 2000

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TWA 1
Wear Test Methods

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April 2000

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3. Activities

The major activities since the last report have been to start and organize three proposed projects. This preparation period revealed that it becomes more and more difficult to find the necessary engagement for VAMAS projects without any financial support due to the change in institutes' policy also for the non-private ones. The draft "VAMAS STRATEGIC PLAN" may help to overcome the weak interest in VAMAS projects.

4. Status of Current Projects

Project No. 1 “Compilation of wear test standards”

Project leader:

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Project objectives:

The objective is to provide a validated source for standardized wear test methods that can be used by engineers and tribologists.

Progress:

Validation of a database of wear testing standards. This database was established as part of a DTI (UK) funded project. It contains over 400 standards. The database will be validated by VAMAS participants based on their expertise and knowledge of the usefulness and robustness of the different documents. Standards that are obsolete will be removed from the database. The value added through the VAMAS work will be comments on the applicability and usefulness of the standard to industry. The preparatory work for this is now complete, with input from experts in Europe and the USA on the most efficient way to proceed. There has already been some enthusiastic response from UK experts about the eventual availability of the validated database.

Standardization activities:

The result will be a database on standardized methods.

Project No. 2 “Ball cratering wear testing”

Project leader:

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Project objectives:

The objective is to perform pre-standardization work in order to establish the “Ball cratering wear testing” method as a standard and to produce a recommended procedure for carrying out the test.

Progress:

Developing the ball cratering test for abrasive wear testing, particularly of coatings. The draft test procedure for this has been written and potential participants identified in the USA and Europe.

Standardization activities:

The project leader has also been appointed as the ASTM task leader for the development of this test, so it is highly likely that this test will lead quickly to the development of a standard.

Project No. 3 "Wear debris characterization methods and representation"
(in conjunction with TWA 7)

Project leader:

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Project objectives:

The objectives are to develop wear debris characterization methods in terms of size, shape, and morphology and develop mathematical representation of the debris.

Progress:

The procedures for wear debris generation are defined for a round robin exercise; the project leader has prepared a survey of methods for wear debris analysis and representation.
The preparation of wear test samples is going on.

Standardization activities:

The friction test conditions were conformed to ASTM F-732

5. Proposed/Future Projects

With regard of the status of the current projects no future projects are seriously discussed.
There exist only Ideas for new future projects are:

- Development of a Certified Reference Material for friction (and wear)
- Calibration of friction measurement on a nano scale

For the successful realization of both projects a third party funding will be necessary.

6. Meetings

No meetings are planed in the next future.

VAMAS Technical Working Area 2

Surface Chemical Analysis

Annual Report

April, 2000

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OVERVIEW

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Information on TWA 2 projects is presented in this report in three groups. In the first group, the objectives and recent work for the 16 current active TWA projects are given. Figure 1 gives a pictorial indication of progress. In the second group, brief accounts are given of the 21 projects that have ended. For two of these projects (projects 1 and 10), some supplementary work has been performed during the past year. Finally, in the final group, there are 3 inactive projects which, after initiation, could not be completed because of lack of resources.

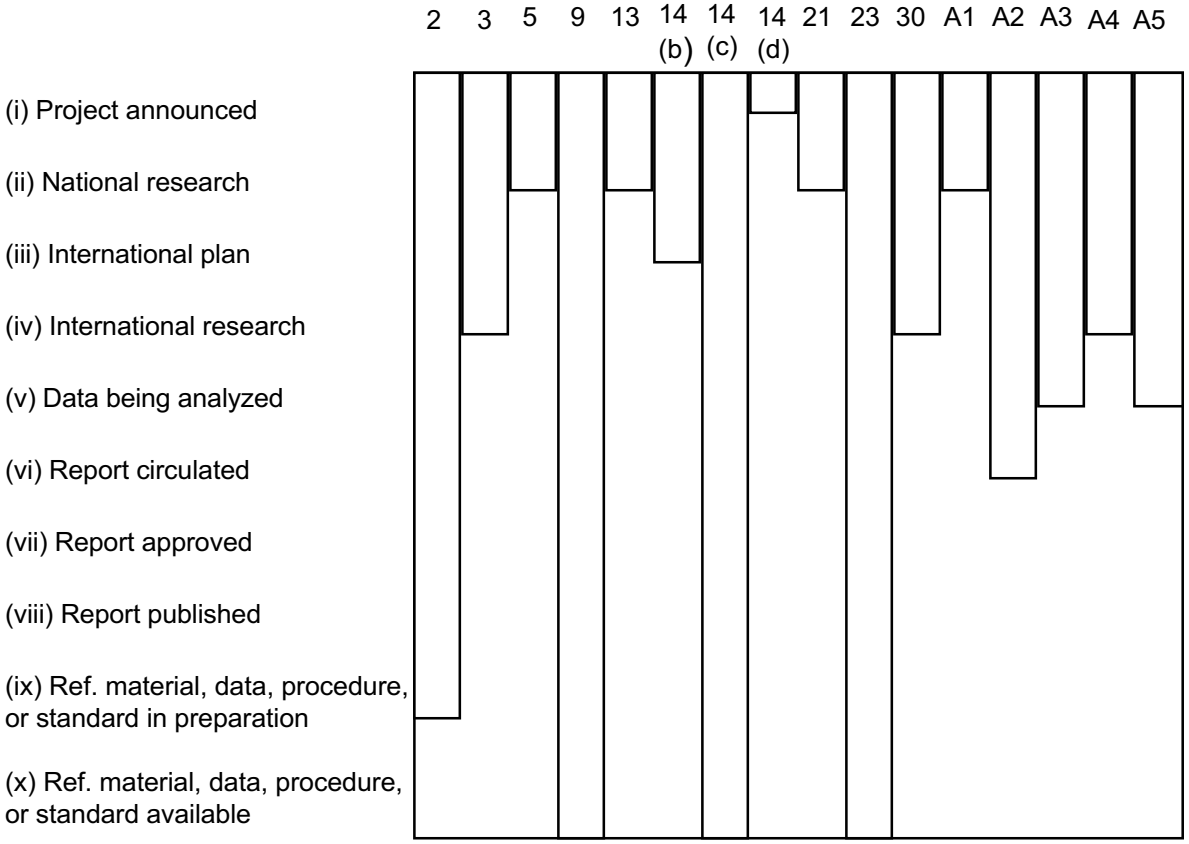

Fig. 1. Diagram showing the progress of TWA 2 active projects.

There has been satisfactory progress in the active TWA 2 projects during the past year. Four interlaboratory comparisons are being conducted (projects A2, A3, A4, and A5) for which there are 26, 18, 36, and 38 participants, respectively, in most of the VAMAS member states. In other projects, improved scientific infrastructure and calibration capabilities are being developed.

Many of the outputs of TWA 2 projects have been incorporated or are in the process of being incorporated into ISO standards, as indicated in Table 1. Table 2 shows the titles of standards or draft standards of ISO Technical Committee 201 on Surface Chemical Analysis arising from or related to TWA 2 projects. TWA 2 is a category-A liaison body with ISO/TC 201 and its subcommittees, and there is excellent communication between these groups.

Table 1. Relationships of VAMAS TWA 2 Projects to ISO Technical Committee 201 on Surface Chemical Analysis.

ISO/TC 201 subcommittee	TWA 2 projects associated with ISO/TC 201 standards	Related TWA 2 projects
SC1: Terminology		
SC2: General Procedures	5	
SC3: Data Management and Treatment	10, 24, 29, 30	13, 21
SC4: Depth Profiling	1, 5	18
SC5: AES	9, 17, 20	4, 13, 14(b), 22, 27
SC6: SIMS		19, A1, A3
SC7: XPS	2, 23, A2, A5	3, 14(a), 14(c), 14(d), 16, 21, 25, 26, 28, 31
SC8: GDS		A4

Table 2. Title of standards or draft standards of ISO/TC 201 arising from or related to TWA 2 projects.

ISO/TC 201 subcommittee	Title of standard or draft standard
SC2: General Procedures	NP 16268: Ion-implanted surface-analytical reference materials—Procedure for standardising the retained areic dose in a working reference material
SC3: Data Management and Treatment	ISO 14976: Surface chemical analysis—Data transfer format DIS 14975: Surface chemical analysis—Information formats WD 15760: Data dictionary for the description of X-ray photoelectron and Auger electron spectroscopy data records
SC4: Depth Profiling	FDIS 14606: Surface chemical analysis—Sputter depth profiling—Optimisation using layered systems as reference materials CD 15969: Surface chemical analysis—Depth profiling—Measurement of sputtered depth
SC5: AES	NP 18118: Surface chemical analysis—Auger electron spectroscopy and X-ray photoelectron spectroscopy—Guide to the use of experimental relative sensitivity factors for the quantitative analysis of homogeneous materials
SC7: XPS	DIS 15472: Surface chemical analysis—X-ray photoelectron spectroscopy—Calibration of energy scales WD 17973: Surface chemical analysis—Medium resolution Auger electron spectrometers—Calibration of energy scales for elemental analysis WD 17974: Surface chemical analysis—High resolution Auger electron spectrometers—Calibration of energy scales for elemental and chemical state analysis WD 18327: Surface chemical analysis—X-ray photoelectron spectroscopy—Guidelines for estimating unintended degradation in a material

Projects 9, 14(c), and 23 have been completed during the past year. The completion of projects 9 and 23 through the provision of a service by the UK National Physical laboratory to calibrate the intensity scales of AES and XPS instruments is a major accomplishment. Early interlaboratory comparisons, conducted some 20 years ago, showed that ratios of measured intensities of different peaks for the same specimen could vary by up to one or two orders of magnitude, for XPS and AES, respectively, on different instruments. Through careful measurements, analyses of instrumental characteristics, comparisons with theory, and additional interlaboratory comparisons conducted with TWA 2, Martin Seah and his coworkers have provided the means to ensure that reliable AES and XPS measurements can be made. The new NPL calibration service developed by Peter Cumpson should have a significant impact on the quality of future surface analyses by AES and XPS. The completion of project 14(c) by Prof. Sven Tougaard of the University of Southern Denmark is also a major accomplishment. Another necessary requirement for a reliable XPS analysis is that the photoelectron intensity be measured and analyzed correctly. For many years, scientists have used simple, empirical methods to measure peak intensities (although it was known that these methods did not have physical justification) and have assumed that the specimen material was homogeneous over the sampling depth to obtain a composition. The latter assumption is often grossly incorrect. Prof. Tougaard and his coworkers have developed and applied a physical model for the transport of the signal electrons in XPS. As a result of this work, it is now possible to obtain critical morphological information for the specimen and to measure the compositions of different phases; software for this purpose is now available. A closely related project to test algorithms for angle-resolved XPS has been recently proposed by Prof. Tougaard (project 14(d)).

The states leading active TWA 2 projects are as follows:

Canada	A1
Germany	5, 13, A2, A4
Japan	30, A5
UK	2, 9, 21, 23, A3
USA	2, 3
CEC	14(b), 14(c), 14(d)

Readers of this report may have noticed that the TWA 2 chairmanship has rotated from NPL to NIST. I would like to express appreciation, on behalf of TWA 2 and myself, to Martin Seah for his leadership of TWA 2 from 1988 through 1999. During this period, the number and impact of TWA 2 projects have grown considerably.

The next meeting of TWA 2 is scheduled to be held on Monday, July 3, 2000 at 9.00 am in Room 30BC20, School of Mechanical and Materials Engineering, University of Surrey, Guildford, UK.

ACTIVE PROJECTS

(a) List of Project Titles and Project Leaders

2.* Development of calibration data for the energy scales of Auger-electron spectrometers (M P Seah, C J Powell)

3. Procedures for quantitative X-ray photoelectron spectroscopy (C J Powell, J E Fulghum)

5.* Development of reference materials prepared by ion implantation (W H Gries)

9.* Intercomparison of Auger-electron energy and intensity measurements (M P Seah)

13. Tests of algorithms for data processing in AES - Factor analysis and intensity (H J Steffen)

14.* (b) Tests of algorithms for background subtraction in AES (S Tougaard)

(c) Tests of algorithms for quantitative XPS by peak and peak-background shape analysis (S Tougaard)
(d) Tests of algorithms for angle-resolved XPS (S Tougaard)

21. Tests of algorithms for the analysis of multicomponent spectra in XPS (A F Carley and M P Seah)

23.* Absolute calibration of XPS instrument intensity scales (P J Cumpson)

30. Development of a Common Data Processing System for AES and XPS (K Yoshihara)

A1. Use of the infinite velocity method for SIMS quantification (N S McIntyre)

A2. The evaluation of static charge stabilisation and determination methods in XPS on non-conducting samples (W Unger, Th Gross, and U Gelius)

A3. Interlaboratory study of static SIMS repeatability and reproducibility (I S Gilmore)

A4. Evaluation of multilayer reference coatings for quantitative GDOES depth profiling (U Beck and Th Wirth)

A5. Interlaboratory study of the degradation of organic materials in XPS analysis (K Yoshihara)

*These projects received support from the Community Bureau of Reference, EC.

(b) Project Status Reports

Project 2: Development of calibration data for the energy scales of Auger electron spectrometers

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Project objective: To develop traceable data for Cu, Ag and Au to calibrate the energy scales of Auger electron spectrometers and to test these data and the relevant protocol for their validity on instruments in the Member States to better than $\pm$3 times the repeatability standard deviation or $\pm$0.2 eV.

Progress since last report: Calculations have been made of the effects of operating conditions on the reference peak energies for the draft ISO standards detailed below.

Standardization activity: Working Drafts of "ISO/NWI 17973: Surface chemical analysis - Medium resolution Auger electron spectrometers - Calibration of energy scales for elemental analysis" and "ISO/NWI 17974: Surface chemical analysis - High resolution Auger electron spectrometers - Calibration of energy scales for elemental and chemical state analysis" have been prepared using the data reported in 1999. These drafts have been circulated to experts and comments have been received which have used to modify the working drafts in order to generate committee drafts for circulation.

Presentation: "Instrument Calibration - Recent Developments," by P J Cumpson on behalf of M P Seah, IUVSTA meeting "XPS: From Physics to Data", Hungary, 26 - 30 April, 1999.

Project 3: Procedures for quantitative X-ray photoelectron spectroscopy

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Project objective: To produce and characterise thin-film structures in order to test electron transport models developed for angle-resolved XPS.

Progress since last report: In collaboration with A. Jablonski (Institute of Physical Chemistry, Polish Academy of Sciences, Warsaw, Poland), results of calculations have been published showing the relationships of the mean escape depth (MED) and the effective attenuation length (EAL) to the inelastic mean free path (IMFP) in angle-resolved XPS (ARXPS). For several representative solids, the ratio of the MED calculated with elastic-electron scattering considered to the MED calculated with elastic scattering neglected varied slowly with electron emission angle for emission angles between zero and 60˚ and then increased rapidly with further increase of emission angle. For emission angles less than about 60˚, it is then possible to use algorithms in current use for the analysis of ARXPS data in which elastic scattering has been neglected. In these analyses, the average EAL should be used as the "lambda" parameter rather than the IMFP; the ratio of the average EAL to the IMFP is simply given by the ratio of the MED obtained with elastic scattering considered to the MED with elastic scattering neglected.

Plans have been made to perform similar calculations for thin films of SiO_2 on silicon. The fabrication of thin gate oxide films with known thickness is important in semiconductor electronics but recent comparisons of measurements by different methods (including XPS) have shown wide variations, sometimes by more than a factor of three. The Si 2p photoelectron intensities, excited by Mg and Al Kα x rays, will be computed for both substrate and oxide. These calculations will be made for different electron emission angles corresponding to those used in ARXPS experiments. It is planned to compare these calculated intensities with corresponding measurements.

Publication: A. Jablonski and C. J. Powell, J. Electron Spectrosc. 100, 137 (1999).

Presentation: C. J. Powell, "Relationships between Parameters Describing Inelastic Electron Scattering in Solids," American Vacuum Society International Symposium, Seattle, Washington, October 27, 1999.

Project 5: Development of Reference Materials prepared by Ion Implantation

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Project objective: To establish a primary system of accurately calibrating dopant levels in semiconductor materials. To establish a system of accurately relating secondary standards to the primary ones and of working standards to those secondary ones to test the results against other reference materials from the Member States by using those as working standards.

Progress since last report: Work in Project 5 is closely tied to work on an ISO Standard, titled *Ion-Implanted Surface-Analytical Reference Material: Procedure for Standardizing the Retained Areic Dose in a Working Reference Material*, which was submitted to ISO TC201/SC2 in December 1997 for member voting as a New Work Item (NWI). Due to a misunderstanding of the voting commitment by some of the members, the necessary votes for acceptance as a NWI were collected only in January 2000. As a consequence, not only work on the Draft Standard but also the associated pre-normative work in Project 5 has slowed down, and, in fact, has been dormant since the last report. Meanwhile, it has been agreed with the chair of ISO TC 201/SC2 that work on the document shall be resumed in the months of April and

May 2000, and 1 June has been proposed as a deadline for submission of an updated document. Consequently, work in Project 5 will resume simultaneously.

Project 9: Intercomparison of Auger electron intensity measurements

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Project objective: To develop a method of calibrating the intensity scale of Auger electron spectrometers and to test this procedure in the VAMAS Member States to better than ±5% with a view to establishing (i) a universal, traceable calibration methodology, and (ii) recommendations for instrument improvement (if necessary) for manufacturers in the Member States.

Progress since last report: The AES and XPS calibration systems are now available at http://www.npl.co.uk/npl/cmmt/sis/index.html.

Presentations: "Instrument Calibration - Recent Developments," by P J Cumpson on behalf of M P Seah, IUVSTA meeting "XPS: From Physics to Data", Hungary, 26 - 30 April, 1999; "XPS Spectrometer intensity-calibration software," by P J Cumpson, ECASIA 99, Seville, Spain, 3 - 8 October 1999.

Project 13: Tests of Algorithms for Data Processing in AES – Factor Analysis and intensity

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Project objective: Development of a reference factor analysis procedure which gives spectral intensities, spectra of unknown components, number of components and allows the identification of contributing factors in the spectral mixture. The factors that influence the accuracy of the procedure should be characterised.

Progress since last report: The computer program in Visual Basic has been completed. A routine was included which creates data sets for testing. The program will be available as a user-friendly version to the public in the coming months and can be downloaded at our new web site: www.materials-science-institute.de. A round-robin study is envisaged after installation.

Project 14(b): Tests of algorithms for background subtraction in AES

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Project objective: To develop algorithms for background correction in AES. To test the validity of these against other methods currently used in Member States.

Progress since last report: The depth excitation function has a significant influence on the shape and relative intensity of the background signal. Therefore, we have started by studying the depth excitation function under varying primary-electron excitation energy. Data analysis has been completed for Si, Fe, Ni, Cu, Ag, and Au and results have been published in refs. [3-5] below. The found depth excitation functions are shallower than had previously been assumed. Monte Carlo simulations have been initiated to gain more insight into the underlying physics (refs. [12, 18] below).

Project 14(c): Tests of algorithms for quantitative XPS by peak shape analysis.

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Project objective: To develop algorithms for quantitative non-destructive analysis of surfaces with sub-nanometer depth resolution. To test the validity of these against other methods currently used in Member States.

Progress since last report: *This project has been completed.* Thus, the level of accuracy of quantitative analysis of surface nano structures by analysis of the XPS peak shape is now largely established and was reviewed in ref. [20]. It has been concluded that information on the concentration depth-profile in the surface region up to depths ~ $5\lambda_i$ is primarily contained in the spectral energy region up to ~ 100 eV below the peak energy. Analysis of a larger energy range than 100 eV does not add much to the information on the details of the structure in the outermost $5\lambda_i$ but gives the possibility to determine additional structural parameters that describe the composition at larger depths. The three most important parameters needed to describe the main characteristics of the distribution of atoms are determined with an uncertainty of typically 5-10 %. The uncertainty in parameters that describe more details of the depth distribution is considerably larger and is typically > 35 %. Different models of depth profiles can be distinguished when they differ significantly over a width of more than ~$1/3\lambda_i$ at any depth < $5\lambda_i$. The uncertainty in the total determined amount of atoms within the surface region is ~ 5-10 % as long as the depths are within the primary probing depth of the method (i.e. < $5\lambda_i$). The absolute quantification of total amounts of atoms is typically accurate to within ~ 15%.

The following four sets of approaches were used to establish this level of accuracy for the technique:

1. Sets of samples were produced for which the total amount of material within the surface region is known to be identical but for which the in depth distribution varies (typically obtained by annealing or consecutive evaporations). Analysis was used to establish the uncertainty with which the technique can determine the amount of substance within the surface region of solids.

2. The growth structure of systems was studied for which independent information from other techniques (in particular AFM) on the growth mechanism is available. We have studied several such systems including metal/metal, metal/silicon, and germanium/silicon systems.

3. Thin films were produced by evaporation and the total amount of deposited material was determined by Rutherford Backscattering. By comparing to the total amount determined by analysis of the XPS peak shape the absolute accuracy for the technique to determine the amount of substance in the surface region was determined.

The following three systems were studied:

(a) thin Ge-films on Si (in collaboration with Aarhus University, Denmark). The results are published in ref.[21]

(b) gold-films on Nickel (in collaboration with C. Jeynes, University of Surrey, England). The results are published in ref.[23]

(c) Copper films on nickel (in collaboration with W.F. Egelhoff and C. J. Powell, NIST, USA) (to be published)

4. Model spectra were calculated and the sensitivity of peak shape to changes in the surface nano-structure was studied to establish the depth resolution of the technique.

Project 14(d): Tests of algorithms for angle-resolved XPS

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Project objective: To establish the validity and accuracy of the ARXPS technique to determine: (1) the amount of substance within the outermost 0-10 nm of a solid; and (2) the in-depth composition profile within the outermost 0-10 nm of a solid

An approach similar to points 1-4 under project 14 (c) will be used. In addition, comparisons will be made of results obtained from ARXPS with results obtained for the same samples from the peak shape analysis technique evaluated in project 14 (c).

Present status of the project: A new software package to analyze ARXPS is presently being developed in *C++* at the University of Southern Denmark. The software has a menu driven graphical user interface that allows interactive change of the in-depth composition profile. It also has optimization routines to carry out automatic search for the best structural parameters. It is planned to include different practical and fast procedures to correct for elastic electron scattering.

Those interested in participating in the intercomparison work are encouraged to contact the project leader.

Papers published since 1995 as the result of VAMAS projects 14(a), 14(b), and 14(c):

1. C. Jansson, S. Tougaard, G. Beamson, D. Briggs, S.F. Davies, A. Rossi, R. Hauert, G. Hobi, N.M.D. Brown, B.J. Meenan, C.A. Anderson, M. Repoux, C. Malitesta, and L. Sabbatini, "Intercomparison of Algorithms for Background Correction in XPS," Surface Interface Analysis, 23, 484-494 (1995)

2. S. Tougaard and A. Jablonski, "Test of Elastic Electron Scattering Corrections for Quantitative XPS," Surface Interface Analysis, 23, 559-564 (1995)

3. D. Fujita, M. Schleberger and S. Tougaard, “Estimation of Excitation Depth Distribution from Electron-excited Auger Spectra of Iron Using Peak-shape Analysis,” Surface and Interface Analysis, 24, 211-216 (1996)

4. D. Fujita, M. Schleberger and S. Tougaard, “Extraction of Depth Distribution of Electron-excited Auger-electrons in Fe, Ni, and Si Using Inelastic Peak-shape Analysis,” Surface Science, 357-358, 180-185 (1996).

5. M. Schleberger, D. Fujita, and S. Tougaard, “The Characteristic Depth of the Excitation Function for Auger electrons in the Noble Metals, Cu, Ag, and Au Determined by Inelastic Background Analysis,” J. Electron Spectroscopy and Related Phenomena, 82, 173-178 (1996).

6. M. Schleberger, D. Fujita, C. Scharfschwerdt and S. Tougaard, “Growth and In-depth distribution of thin metal films on silicon (111) studied by XPS- Inelastic Peak Shape Analysis,” Surface Science 331-333, 942-947 (1995).

7. F. Yubero, C. Jansson, D.R. Batchelor, and S. Tougaard, “Validity of method for quantitative XPS of surface nano-structures determined by application to Cu/Au/Cu,” Surface Science 331-333, 753-758 (1995).

8. D. Fujita, M. Schleberger and S. Tougaard, “XPS Study of the Surface Enrichment Process of Carbon on C-doped Ni(111) Using Inelastic Background Analysis,” Surface Science 331-333, 343-348 (1995).

9. M. Schleberger, D. Fujita, C. Scharfschwerdt and S. Tougaard, “Nano-Structure of Thin Metal Films on Silicon (111) Investigated by XPS: Inelastic Peak Shape Analysis,” Journal Vacuum Science Technology, B13., 949-953 (1995).

10. S. Tougaard, “Quantitative XPS: Non Destructive Analysis of Surface Nano-Structures,” Applied Surface Science, 100/101, 1-10 (1996).

11. S. Tougaard, “Surface Nano-structure Determination by XPS Peakshape Analysis,” J. Vacuum Science Technol. , A14, 1415-1423 (1996).

12. A. Jablonski and S. Tougaard, “The Excitation Depth Distribution Function for Auger Electrons Created by Electron Impact,” Surface Interface Analysis, 25, 688-698 (1997).

13. S. Tougaard, "Background Subtraction for Quantitative XPS", Journal of Surface Analysis, 1, 153-156 (1995).

14. S. Tougaard, “Universality Classes of Inelastic Electron Scattering Cross Sections,” Surface Interface Analysis, 25, 137-155 (1997).

15. I. S. Tilinin, A. Jablonski, and S. Tougaard, “Emission-depth Dependence of the Signal Photoelectron Energy Spectrum,” Surface Interface Analysis, 25, 119-131 (1997).

16. S. Tougaard and A. Jablonski, “Quantitative XPS: Influence of Elastic Electron Scattering,” Surface Interface Analysis 25, 404-408 (1997).

17. A. Cohen Simonsen, F. Yubero, and S. Tougaard, “Quantitative Model for Electron Energy Loss in XPS,” Phys. Rev. B 56, 1612 (1997).

18. A. Jablonski and S. Tougaard, “Practical Correction Formula for Elastic Electron Scattering Effects in Attenuation of Auger- and Photo- Electrons, Surface Interface Analysis, 26, 17 (1998).

19. M. Schleberger, A. C. Simonsen, S. Tougaard, J. L. Hansen, and A. Nylandsted Larsen, “Ge Growth on Si(001) studied by XPS Peak shape analysis and AFM,” Journal Vacuum Science Technology A 15, 3032-3036, 1997.

20. S. Tougaard, “Accuracy of the Non-destructive Surface Nano-structure Quantification Technique based on Analysis of the XPS or AES Peakshape,” Surface Interface Analysis, 26, 249-269, 1998.

21. A. C. Simonsen, M. Schleberger, S. Tougaard, J. L. Hansen, and A. Nylandsted Larsen, “Nano-structure of Ge deposited on Si(001): A study by XPS Peak shape analysis and AFM”
Thin Solid Films, 338, 165-171, 1998.

22. A. Jablonski and S. Tougaard, “Evaluation of Validity of the Depth-dependent Correction Formula (CF) for Elastic Electron Scattering Effects in AES and XPS,” Surface Interface Analysis, 26, 374 – 385, 1998.

23. A. Cohen Simonsen, J. P. Pøhler, C. Jeynes, S. Tougaard, “Quantification of Au deposited on Ni: XPS Peak Shape Analysis Compared to RBS,” Surface Interface Analysis, 27, 52-56, 1999.

24. A. Jablonski and S. Tougaard, “Escape Probability of Electrons from Solids. Influence of Elastic Electron Scattering,” Surface Science. 432, 211-227 (1999).

25. C. J. Powell, J. R. Rumble, D. M. Blakeslee, M. E. Dal-Favero, A. Jablonski, and S. Tougaard, “The NIST Surface Analysis Center,” p. 887- 891 in “Characterization and Metrology for ULSI Technology”, D. G. Seiler et al. (Eds.), The American Institute of Physics, 1998.

26. K. M. Krawczyk, A. Jablonski, S. Tougaard, J. Toth, D. Varga, and G. Gergely, ”The Inelastic Mean Free Path and the Inelastic Scattering Cross-section of Electrons in GaAs Determined from Highly Resolved Electron Energy Spectra,” Surface Science, 402-404, p. 491-495 (1998).

27. A. Cohen Simonsen, F. Yubero, S. Tougaard, “Analysis of angle resolved electron energy loss in XPS spectra of Ag, Au, Co, Cu, Fe, and Si,” Surface Science 436, 149-159(1999).

28. A. Cohen Simonsen, S. Tougaard, J. L. Hansen, and A. Nylandsted Larsen, "Temperature Dependent Morphology of Oxidized Ge films on Si(001): XPS peak shape analysis and AFM,"
Surface Interface Analysis, 2000 (submitted).

29. A. Dubus, A. Jablonski, and S. Tougaard, "Evaluation of Theoretical Models for Elastic Electron Backscattering from Surfaces," Progress in Surface Science, 2000, in press.

The projects are partially supported by the EEC, Community Bureau of References (BCR) and the Danish Natural Research Science Foundation.

Project 21: Tests of algorithms for the analysis of multicomponent spectra in XPS

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Project objectives: To investigate and compare algorithms for the analysis of X-ray photoelectron spectral envelopes comprising several overlapping components. Two types of data will be examined - individual 'one-off' spectra, and sets of related spectra, such as those obtained in a sputter-profiling experiment. Initially, synthesised data will be used, but the eventual aim will be to provide standardised real composite spectra for analysis. Of key interest will be the ability of a particular algorithm to (a) deduce the number of components present (b) estimate the peak parameters, such as height, FWHM, position and asymmetry (c) provide an estimate of the errors in the computed parameters (d) cope with an increasing degree of overlap. As well as testing those algorithms 'conventionally' applied to XPS data, exploring numerical approaches from other spectroscopies could provide especially productive. The long-term objective is to produce a set of well-characterised public domain programs, with the aim of standardising the methods used in this area of data analysis.

Progress since last report: The test data used for this study, reported in J Elec Spec **95** (1998) 71-93 are now available for manufacturers and software writers to test their software.

Project 23: Absolute calibration of XPS instrument intensity scales

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Project objective: To develop an absolute calibration procedure with which to calibrate the absolute intensity/energy transmission function of all XPS spectrometers used in Member States (large area, small spot, imaging, monochromated, unmonochromated, Al or Mg radiation) to better than $\pm$5% over the energy range 100 to 1500 eV.

Progress since last report: The AES and XPS calibration systems are now available at http://www.npl.co.uk/npl/cmmt/sis/index.html.

Presentations: "Instrument Calibration - Recent Developments," by P J Cumpson on behalf of M P Seah, IUVSTA meeting "XPS: From Physics to Data", Hungary, 26 - 30 April, 1999; "XPS Spectrometer intensity-calibration software," by P J Cumpson, ECASIA 99, Seville, Spain, 3 - 8 October 1999.

Project 30: Development of a Common Data Processing System for AES and XPS

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Project objective: To construct software in Quick Basic for IBM PC or NEC PC with MS-DOS to manipulate AES and XPS spectra in a standard manner. To discuss requirements and coordinate contributions from Member States.

Progress since last report: The software named Common Data Processing System that runs on Windows has been developed. The Common Data Processing System(COMPRO) is designed to be a program to assess the data-processing procedure provided by scientists, to check a spectrum, and to build both spectral and physical property databases. The present version of COMPRO is 6.44, and it includes a sensitivity database for AES and XPS in addition to the existing databases. COMPRO version 6.44 can be downloaded from http://sekimori.nrim.go.jp.

Standardization activities: COMPRO can convert original AES and XPS data files to ISO 14975 and 14976 standard formats.

Publications:
K. Yoshihara and M. Yoshitake, J. Vac. Sci. Technol., A16, 1388 (1998).
K. Yoshihara, J. Surf. Anal. 5, 98 (1999).

Project A1: Use of the infinite velocity method for SIMS quantification

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Project objective: The general evaluation of a standardless quantification method (the infinite velocity model) for dynamic SIMS. The will be carried out by evaluating the random and systematic variations of concentration profiles (calculated via the infinite velocity method) collected from: (i) different laboratories (operators), (ii) different instruments (limited to the Cameca IMS-3f, 4f, 5f and 6f group of instruments, since these are the only instruments for which the transmission function has been defined).

Progress since last report: Partly as a result of a recent detailed critical evaluation [1] of the Infinite Velocity (IV) approach, we have begun to examine in detail some of the cases where IV-based quantitation is known to exhibit particularly poor agreement with the known composition.

Gallium arsenide (GaAs) was chosen for a particularly exhaustive investigation because, in addition to its relatively poor agreement with expected composition in initial IV trials, this material in available in high purity and the mass differences are small. Primary ion bombardment with several keV $^{40}Ar^{+}$, $^{16}O_2^{+}$, $^{16}O^{+}$, $^{16}O^{-}$ and $^{133}Cs^{+}$ was applied in order to compare positive and negative ion emission from sputter-cleaned GaAs surfaces with that from slightly oxidized or slightly cesiated surfaces. Kinetic energy distributions have been obtained for both positively- and negatively-charged secondary ions of $^{69,71}Ga^{+,-}$ and $^{75}As^{+,-}$ and others, such as $^{133}Cs^{+}$ and $^{16}O^{-}$. The kinetic energy range investigated extended in all cases to 700 eV, and in some cases to several keV. For clean GaAs (Ar^{+} bombardment), Ga/As was overestimated by a factor of 60 using positive secondary ions and As/Ga by a factor of 10 using negative secondary ions. For an oxidised surface (O_2^{+} or O^{-} bombardment), Ga/As was overestimated by a factor of 15 for positive ions and 3 for negative secondary ions. For a caesiated GaAs surface, the As/Ga ratio for negative secondary ions was 20. The better agreement for negative secondary ions may be related to a smaller difference in electron affinity than ionisation potentials for these particular elements.

Zinc and copper also have large differences in their ionisation potentials; therefore IV measurements are being undertaken on two CuZn alloys of known composition. For the NIST CuZn alloy (SRM 1112), on a clean surface (Ar^{+} bombardment) the Zn composition is overestimated by a factor of 7 for positive secondary ions, while on an oxidised surface (O^{-} bombardment) for positive secondary ions, Zn is overestimated by a factor of 2. Experiments have started on beta brass (Cu/Zn of 0.81) to see if similar overestimation factors are found.

It is clear that at least some IV-determined quantitative results need to be “corrected” with experimentally-determined factors. Whether the magnitude of such a factor can be anticipated from ionisation potentials or electron affinities is not yet known.

[1] K. Wittmaack, Surf. Sci. 429 (1999) 84.

Project A2: Evaluation of static charge stabilization and determination methods in XPS on non-conducting samples

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Project objective*:* To provide reference procedures for surface charge stabilisation and uniformation to a defined potential to be used in practical surface analysis with XPS.

Progress since last report: The final report on the inter-laboratory comparison (list of participants, see above) is written now. It will be distributed to the participants on 1 April.

Standardization activities: The surface charge has to be determined in XPS analysis on insulating samples to provide chemical state data to be used for data banks and elemental identification. Standards cannot be written without a valid procedure and reference data. At present no ISO or CEN standards exist. The standards would be used for research, product development and quality control in chemicals, electronics and engineering industries. ASTM is already involved (Guide N° E 1523-93), and ISO will be involved.

Talks, reports and publications:

W. E. S. Unger, O. Böse, Th. Gross, A. Lippitz, U. Gelius, "Results of a VAMAS TWA 2 inter-laboratory test experiment: Evaluation of static charge stabilization and determination methods in XPS on non-conducting samples," ECASIA '99, Sevilla, 4-8 October 1999

"Project A2: Evaluation of static charge stabilization and determination methods in XPS on non-conducting samples: Report on an inter-laboratory comparison," Report, BAM, Berlin, March 2000.

Project A3: Interlaboratory study of static SIMS repeatability and reproducibility

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Project objectives: To study the repeatability and reproducibility of static SIMS studies on conductors and insulators.

Progress since last report: A detailed analysis has been made of all the parameters that may affect the interlaboratory data described in the 1999 report. The results show that the working practice in some laboratories leads to significant contamination but they also show that other laboratories can obtain repeatabilities better than 2% for the bulk polymer and irganox or silver samples. Relative intensities of daughter peaks can be obtained with accuracies better than 3% between laboratories. The effects of different source ions and their energies is now resolved and material is being prepared to propose a protocol for a second inter-laboratory study. Good transferability of data is important for the effective use of reference SSIMS libraries.

Publication: I S Gilmore and M P Seah, "Static SIMS: An Inter-laboratory Study," in Secondary Ion Mass Spectrometry, SIMS XI, Eds G Gillen, R Lareau, J Bennett and F Stevie, Wiley, Chichester (1998), p. 999 - 1002.

Presentation: I S Gilmore, J E Johnstone and M P Seah, "Static SIMS: an evaluation of the effects of ion species and energy and their relation to molecular structure," ECASIA 99, Seville, Spain., 3 - 8 October 1999.

Project A4: Evaluation of multilayer reference coatings for quantitative GDOES depth profiling

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Project Objectives: To provide multilayer reference coating systems and to improve quantitative GDOES depth profiling of coated surfaces.

Progress since last report: The Ti/Al and SiO_2/TiO_2 test samples were distributed to the participants in the Round Robin analysis by the end of April 1999. In December of last year we received the first results. However, because of delayed incoming data we have decided to prolong the final deadline for finishing the measurements to the end of April. We have written a letter to all participants to advise them of this deadline. Currently the evaluation of the results is in progress. The report will be distributed in December of this year.

Project A5: Interlaboratory study of the degradation of organic materials in XPS analysis

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Project objective: To develop a procedure to estimate the degradation by X-ray irradiation during analysis and to develop the possibility of using the degradation behavior to estimate the X-ray source strength.

Progress since last report: We sent NC+CA, PVC and PTFE specimens to 42 institutes in January 1999. 35 institutes (39 laboratories) from Japan, UK, US, Germany, Italy, Switzerland, Korea have returned data. All spectra have been converted to ISO 14976 SDP files by COMPRO. We calculated the peak intensity changes of N of NC+CA, Cl of PVC, and F of PTFE without peak fitting, and got degradation rates (k) by assuming d[A]/dt = -kA (A: amount of polymer). A strong correlation is found between the degradation rate of NC+CA and PVC in case of achromatic X-ray sources. However, in case of chromatic X-ray source, we can get poor correlation between the degradation rate of NC+CA and PVC. The degradation behavior of PTFE was complicated, and we could not find correlation with degradation rates of other polymers.

Standardization activities: A new work item (Surface chemical analysis - X-ray photoelectron spectroscopy - Guidelines for estimating unintended degradation in a material) has been proposed to ISO/TC201. The round robin results will be utilized in this new work item.

Publication: A. Tanaka, R. Oiwa and K. Yoshihara, J. Surf. Sci. Soc. Jap., **20**, 791 (1999).

PREVIOUS PROJECTS

(a) List of Project Titles and Project Leaders

1.* Development of thin oxide films as reference materials (M P Seah)

6. XPS intensity calibration and stabilisation with polymeric reference materials (C E Bryson)

7. Correction methods for backscattering in AES (J P Langeron)

8.* Reference data for sputtering rates in oxides (H J Grabke)

10. Development of a standard data transfer format (M P Seah)

11.* Multitechnique characterisation of vacancies in alumina (C Le Gressus)

14 (a)* Tests of algorithms for background subtraction in XPS (S Tougaard)

16.* Intercomparison of surface analysis of thin aluminium oxide films (P Marcus)

17. Quantitative AES of Au/Cu alloys (R Shimizu)

18. Evaluation of LOGIT, an algorithm for fitting sputter-depth-profile data, for the measurement of interface widths of a NIST thin-film reference material (J Fine)

19. Round Robin SIMS study of impurities in GaAs crystals (S Kurosawa)

20. Round Robin AES study of Co-Ni alloys (K Yoshihara)

22. Calibration of channel electron multiplier detection efficiency stabilities (M P Seah)

23. Absolute calibration of XPS instrument intensity scales (P J Cumpson)

24. Conventions for spectral data bases (R N Lee)

25. Quantitative XPS of Au-Cu alloys (K Yoshihara)

26. Theoretical assessment of escape depth (R Shimizu)

27.* Multiline reference material for differential AES intensity calibration (M P Seah)

28. Quantitative XPS of Co-Ni alloys (K Yoshihara)

29. Development of a File Format Transition System (K Yoshihara)

31.* Intercomparison of the effects of attenuation length on the determination of thin oxide film thicknesses (P Marcus)

*These projects received support from the Community Bureau of Reference, CEC.

(b) Project Information

A brief summary is given here of TWA 2 projects that have ended; more details can be found in earlier Annual Reports. In two cases (projects 1 and 10), there have been recent developments and these developments are summarized here.

Project 1: Development of Thin Oxide Films as Reference Materials

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Project objective: To develop and produce certified reference materials, with validation through laboratories in the Member States experienced in the growth of Ta_2O_5 and in measuring oxide

thicknesses to define (i) the sputtering rate of argon ion guns, to better than ±3%, and (ii) the instrumental depth resolving capability to better than ±2%.

Progress since last report: The interlaboratory study is complete but material characterisation continues. A new batch of material has been made and the shelf life of existing material checked. The material remains satisfactory. Analysis by AES shows that profiles made in the continuous mode by sputtering whilst recording data leads to O^- ions being detected in the spectrometer over a wide energy range. These can be used for the signal for profiling with the electron beam turned off. Measurements using ToF SIMS shows that this single layer material may be used for defining depth resolution in SIMS very successfully. A depth resolution of 1.0 nm at 30 nm depth is attained with a decay length of 0.3 nm.

Standardisation activity: The ISO standard that this material would be used with is ISO/FDIS 14606 Surface chemical analysis - Sputter depth profiling - Optimization using layered systems as reference materials.

Publication: M P Seah, S J Spencer, I S Gilmore and J E Johnstone, "Depth resolution in sputter depth profiling - characterization of a third batch of tantalum pentoxide on tantalum certified reference material by AES and SIMS," Surf. Interface Anal. **29**, 73-81 (2000).

Presentation: M P Seah, S J Spencer, I S Gilmore and J E Johnstone, "Characterisation of a sputter-depth-profiling certified reference material CRM 261 T," ECASIA 99, Seville, Spain, 3 - 8 October 1999.

Project 6: XPS intensity calibration and stabilisation with polymeric reference materials

Polymers are often studied by XPS and involve the problem of charge neutralisation/stabilisation. Here, putative reference materials have been fabricated with transport containers for interlaboratory test in 6 laboratories in the US, UK and Belgium. It was intended that, reference materials would be established and a second project spawned on charge stabilisation methods. Three Member States were involved (US, UK, B) but effort has now transferred to the parallel project A2. This project is now terminated.

Project 7: Correction methods for backscattering in AES

Backscattering corrections in AES are vital for quantification. Monte Carlo calculations were in progress to assess data for archetypal systems for test. However, the laboratory piloting this work in France has failed to get resource continuity and so this project has stalled. The software is available and discussions have been held with scientists in France, Japan and the UK to continue the project. No Member States currently involved. This work is now terminated.

Project 8: Reference data for sputtering rates in oxides

Oxides of relevant metals have been made with defined quantities of material to establish sputtering rates and to see the extent to which thermal oxides agree with sputter deposited films. A limited number of reference samples have been developed but results have not been promising. Oxides are highly relevant in two very different areas (i) thermally for oxidation, corrosion and passivation and (ii) by sputter deposition for optical and electronic devices. The work reached a stage of completion in one Member State (G) but no further work is proposed. This work is now terminated.

Project 10: Development of a standard data transfer format

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Project objective: To develop a general format, with agreement through the Member States, for the transfer of data files from the data capture computers used with surface analysis instruments to other computers for analysis.

Progress since last report: This project is complete, as originally envisaged, with the production of ISO 14976. This is a modular format and so is expandable. In order to accommodate useful information for data basing, information formats have been developed. This year a new information format for SIMS mass calibration has been proposed to ISO/TC 201 to extend ISO 14976.

Standardisation activities: This project led to ISO 14976, Surface chemical analysis - Data transfer format. Information formats for AES and XPS were added as ISO/DIS 14975, Surface chemical analysis - Information format. Static secondary ion mass spectrometry has been proposed with a WD.

Project 11: Multitechnique characterisation of oxygen vacancies in alumina

This work shows the importance of defects in dielectrics and their role in the surface charging of insulators. The national Research programme in France will continue in the area of defect analysis in wear, breakdown, adhesion etc. in ceramics. The general practical problem of charging does not seem fully resolved although a number of separate approaches are now possible. It is not clear if the physics will allow a full universal solution to the charging problem although strong progress is continuing in the materials science problems. This project is now terminated in its present form but the general problem of charge centres in insulators is under active study. This work involved one Member State (F) and is now terminated within this programme.

Project 14(a): Tests of algorithms for background subtraction in XPS

This work is crucial to quantification in XPS and also AES. A large amount of excellent work has now been completed in one Member State and the project has been subdivided into three separate objectives. In part (a) an intercomparison study has been undertaken for elemental samples where data have been recorded in 8 laboratories in UK, I, CH, F, DK and Northern Ireland. Part (a) is complete.

Project 16: Intercomparison of surface analysis of thin aluminium oxide films

A study of many laboratories in four Member States showed that ARXPS could usefully define oxide thicknesses if the relevant attenuation lengths could be calibrated by correlation with NRA. This work showed that the methodology was appropriate and led to a new, refined, project 31. This work is now complete and involved five Member States (F, G, I, CEC and others).

Project 17: Quantitative AES of Au/Cu alloys

This study in Japan showed clearly the need to calibrate each spectrometer individually by using reference materials close to the analyte in composition. This work is finished and led to the setting up of projects 25 and 28. This work involved one Member State (J) and is now complete.

Project 18: Evaluation of LOGIT, an algorithm for fitting sputter-depth-profile data, for the measurement of interface widths of an NBS thin film reference material

Interface shapes in sputter-depth profiling are traditionally characterised by one parameter. This work allows a more detailed characterisation with least squares fitting and uncertainties, so improving the measurement methodology. The study, which has been very successful, is complete. This work involved four Member States (US, C, G, J).

Project 19: Round Robin SIMS study of impurities in GaAs crystals

This study seeks to define consistency and traceability in SIMS studies of dopants using relative sensitivity factors (RSFs) in GaAs. Reference samples have been prepared with different levels of 9 dopants. 31 sets of samples have been analysed in four Member States (J, US, UK, G). Results show that RSFs vary by an order of magnitude with ion angle of incidence. Typical relative standard deviations were 30% but some instruments were significantly worse than others. All work has been published and this work is complete.

Project 20: Round Robin AES study of Co-Ni alloys

This study parallels that of project 17 but has made use of projects 29 and 30 in intercomparisons of data from 19 institutes in Japan. This project involved one Member State (J). This project is complete.

Project 22: Calibration of channel electron multiplier detection efficiency stabilities

Calibrations established in project 9 were re-appraised after 2 years as theoretical predictions indicated that AES instrument calibrations would drift. Some did and some did not. Depending on the type of use, the calibration interval may be set at periods between 1 month and 1 year. The work involved six Member States (UK, C, G, J, USA, CEC) and will eventually be part of documentary standards. This work is now complete.

Project 23: Absolute calibration of XPS instrument intensity scales

This study exactly parallels project 9 but for XPS. The status is as for project 9 and involved all Member States (UK and all States). The work is complete and a traceable calibration service is now available.

Project 24: Conventions for spectral data bases

Conventions for data bases are important so that data can be usefully and reliably stored and retrieved. This project, which is led by the US, is part of a larger effort in ASTM E49 and the journal *Surface Science Spectra* of the American Vacuum Society. Participants in Member States have seen early versions of the documentation and their comments have been included. A working draft is now under consideration by ISO/TC 201/SC3 on Data Management and Techniques. This project is now terminated.

Project 25: Quantitative XPS of Au-Cu alloys

Project 17 is a forerunner to this project that follows the same route. All the reference material is now consumed. One Member State was involved (J). This project is complete.

Project 26: Theoretical assessment of escape depth

There is a great need for a general understanding of the role of elastic scattering in electron spectroscopy. This project was initiated by and involved Japan (J), but is now terminated. Further work on this topic is being conducted in project 3.

Project 27: Multi-line reference material for differential AES intensity calibration

Project 9 homogenised AES intensities from different instruments and allowed them to be traceable to absolute standards. That project was for AES in the direct spectral mode. Older analysers only work in the differential mode so a reference sample was made to allow those instruments to be calibrated traceably to the instruments used for direct spectral analysis. The project met its targets, the work is complete, and a reference material is available. This project involved three Member States (UK, I, G).

Project 28: Quantitative XPS of Co-Ni Alloys

This project follows projects 17 and 25. Results have been acquired from 20 institutes in Japan, and the spectra have been analyzed with COMPRO (project 30) for instrumental transmission and background subtraction. This project has involved one Member State (J), and is now terminated.

Project 29: Development of a file format translation system

The software, Visual Basic for IBM-PC or NEC-PC with Windows to translate spectral data from JEOL, RIGAKU, Surface Science Instruments, Scienta, VG and VSW instruments, has been completed. From the "Windows" version, the conversion of the data file written in binary code is no longer supported. The

manufacturers will be advised to convert their data to text file. The new version supports the conversion of original text file to the ISO 14975 and 14976 formats, and has been incorporated into the Common Data Processing System (Project 30). This project is now terminated.

Project 31: Intercomparison of the Effects of Attenuation Length on the determination of thin oxide film thicknesses

The aims of this project are: (1) to estimate the applicability and uncertainty associated with the exponential law for the attenuation of photoelectrons, and (2) to determine the attenuation lengths of photoelectrons in aluminium and in aluminium oxide.

INACTIVE PROJECTS

The following projects were approved by TWA 2 but no progress has been possible because the needed resources were not available.

4. Measurement of spatial resolution in AES

12. Calibration of surface layers by nuclear reaction analysis

15. Evaluation of SIMS sensitivity factors

FUTURE NATIONAL AND INTERNATIONAL MEETINGS RELEVANT TO TWA 2 ACTIVITIES

The following national and international meetings relevant to TWA 2 activities are scheduled in 2000 and 2001. Further information on each meeting can be obtained from the indicated Internet address or meeting organizer.

International Conference on Quantitative Surface Analysis (July 3-7, 2000), University of Surrey, Guildford, UK (www.surrey.ac.uk/MME/QSA/index.html)
International Conference on Electronic Spectroscopy and Structure (August 7-11, 2000), University of California at Berkeley, USA (www-als.lbl.gov/icess)
SIMS Europe 2000 - 2nd European Workshop on Secondary Ion Mass Spectrometry (September 17-19, 2000), University of Muenster, Germany (organizer: Prof. A. Benninghoven, Physics Department)
International Symposium of the American Vacuum Society (October 2-6, 2000), Boston, USA (www.vacuum.org)
Asia-Pacific Surface and Interface Analysis Conference (October 23-26, 2000), Beijing, China (www.icas.ac.cn/~stm/apsiac.htm)
ECASIA'01 - 9th European Conference on Applications of Surface and Interface Analysis (October 1-5, Avignon, France (www.enscp.jussieu.fr/ECASIA/)
Ninth Topical Conference on Quantitative Surface Analysis (October 26-27), location near San Francisco, USA to be determined (www.vacuum.org)
International Vacuum Congress, International Conference on Solid Surfaces, and International Symposium of the American Vacuum Society (October 29-November 2, 2001), San Francisco, USA (www.vacuum.org)
SIMS XIII - 13th International Conference on Secondary Ion Mass Spectrometry (November 11-16, 2001), Nara, Japan (momiji.esc.u-tokyo.ac.jp/sims13)

TWA 3
Ceramics for Structural Applications

Chairperson: G. Quinn, NIST, USA

Annual Report: March 6, 2000

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General Overview

The round robin on **Fracture Toughness by the SEVNB method** was completed and a comprehensive final report was published. A round robin on **Elevated Temperature Flexural Strength** testing is underway and proceeding satisfactorily. A new round robin on **Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite** is at an advanced state of planning and a proposal may be ready for submission to the Steering Committee. The attached figure illustrates the fourteen projects that have been or will be conducted by TWA 3.

A general overview article: “VAMAS at Twelve” was published by the American Ceramic Society Bulletin in July, 1999. It summarized the 12 round robins and 12,000 experiments that have been completed in VAMAS TWA 3 in the last 12 years.

The TWA last met in June 1999 at the European Ceramic Society Conference in Brighton, England. Seventeen members or their alternates attended. There are no plans to meet in 2000.

The list of standards that have been impacted by TWA 3 projects is growing. This information is now shown on the TWA 3 web site. The current project, fracture toughness by SEVNB, has validated and refined a method to the point that it now is a leading candidate for standardization in CEN TC 184, even though the method was virtually unknown 5 years ago.

Coordination with ISO TC 206, Fine Ceramics has been excellent at the current B liaison level.

Five more potential projects have been identified. Arrangements have been made for the transfer of the chairmanship of this TWA.

Status of Current Projects

1. The ***Fracture Toughness by the SEVNB method round robin*** was completed and a splendid final report prepared the Swiss Federal Laboratories for Materials Testing and Research, Zurich (Mr. Jakob Kübler. The project was very successful and the method will be included in the new European Standards Committee TC 184 standard test method for determination of fracture toughness. The simplicity of the precracking procedure (a common industrial razor blade with diamond paste) appealed to many users particularly in the industrial sector. This project was a joint VAMAS/European Structural Integrity Society project with 35 participating laboratories.

2. The ***High Temperature Flexure Strength*** project began in October. Thirteen laboratories are participating. This project was organized by Dr. M. Mizuno of the Japan Fine Ceramic Center and is intended to support the development of an ISO TC 206, Fine Ceramics, elevated temperature flexure strength test standard. Either 10 x 30 mm or 20 x 40 mm fixtures may be used. Fixtures may either be fully- or semi-articulated. Unfortunately only 12 specimens per lab were available, so statistical fluctuations may be large which may hamper making conclusions. One laboratory (NASA-Glenn) requested and received two sample sets so that they can test 12 specimens with a semi articulating jig and another 12 specimens with a fully articulating jig for a direct comparison. JFCC will do one set of 12 specimens with 10 x 30 mm fixtures and another set of 12 specimens with 20 x 40 mm fixtures.

3. A new project ***Determination of Phase Composition and Percent Crystallinity in Hydroxyapatite*** has been proposed by the National Physical Laboratory in cooperation with Queen Mary College. Hydroxyapatite (HA) is a ceramic material increasingly used as a biocompatible monolithic or coating encouraging adaptation of an implant into the human body, but the body's response depends critically on the phase composition of the material. The objective of this round robin is to assess the level of accuracy and repeatability achievable by a method proposed for the quantification of HA crystallinity and phase composition. Calibration specimens and specimens composed of mixtures of Ca-PO_4 glass, HA, CaO, α-TCP (tri-calcium phosphate) and β-TCP will be supplied for analysis. For parties interested in full participation (including producing a glass powder for preparing the calibration specimens) HA, CaO, α-TCP and β-TCP powders for preparing calibration specimens and specimens composed of mixtures of Ca-PO_4 glass, HA, CaO, α-TCP and β-TCP will be supplied for analysis.

The primary VAMAS output will be a report detailing the findings of the round robin, and giving an assessment of the reliability of the method proposed for the International Standard. The primary standardization output will be a statement of confidence in the technique written into the standard based on the findings of the round robin. Two International Standards for hydroxyapatite ceramics and hydroxyapatite coatings are currently at the committee draft stage of ISO/TC150/SC1. The purpose of these two documents is to specify the key characteristics that define hydroxyapatite materials and to stipulate the requirements that shall be met to allow their use as materials for surgical implants.

PROPOSED FUTURE PROJECTS

The following projects have been proposed and are at an early planning/discussion phase.

1. Surface Roughness

Dr. R. Morrell of the National Physical Laboratory reported that the Phase I, European part of this project was still underway and would need to be analyzed before a joint VAMAS/CEN Phase II project could begin.

Status of Project: On hold pending analysis of Phase I project.

2. Thermal Shock

Dr. R. Wäsche of BAM, Berlin proposed that TWA 3 start a thermal shock project. BAM is constructing a laser shock apparatus for thermal shock evaluation of ceramics. It is understood that few other laboratories could have such equipment, but Dr. Wäsche has proposed that laboratories could compare different methods on a common material. Although our past programs have all been classic interlaboratory round robins, Mr. Quinn agreed that Dr. Wäsche's suggestion is within the scope of the VAMAS Project. Some possible ideas for the project are to:

a. Compare laser shock testing to thermal shock by air or water quenching test methods. (between laboratory comparisons).
b. Compare cyclic thermal fatigue results by different methods (between laboratory comparisons).
c. Compare thermal shock experimental data generated in one or more laboratories to theoretical/modeling analyses prepared by other laboratories.
d. Compare thermal shock results from flexure specimens (rods or rectangular bars) to disk specimens.

Some members feel that suggested that thermal shock equipment is very expensive and laser equipment is not commonly used by industry. Perhaps the laser heating methods could be compared to gas torch or infra-red lamp heating devices. Several other TWA members were worried that not many laboratories could participate in this project. Mr. Quinn believes that some cooperative VAMAS work (other than a classic round robin) could be beneficial and should be explored.

Status of Project: Discussion phase. Project to be developed and defined.

3. Nondestructive Characterization

Dr. O. Toft Sorenson of RISOE, Denmark made a presentation for a new project for nondestructive characterization (NDC) of ceramics. This project would be a collaboration with Dr. Joergen Rheinländer and Dr. Bent Sorenson at RISOE The objective is to determine whether micro-defects in ceramics can be characterized effectively by current nondestructive characterization techniques and whether the data obtained by these techniques can be used reliably to predict mechanical strength.

RISOE is able to prepare specimens with seeded (intentionally created) flaws. The specimens (presumably flexure specimens although other types could be considered) would be distributed to the round robin participants who would try various NDC methods such as x-ray microfocus, ultrasonic scanning, etc. Specimens would then be tested to fracture and fractographic analysis conducted to confirm the fracture origin was the seeded flaw type.

Status of Project: Discussion phase. Project to be defined and developed.

4. Elastic Modulus

Dr. R. Morrell of NPL suggested a possible project for elastic modulus determination. The project would include: beam static flexure, beam resonance by forced excitation, beam resonance by impulse excitation, and ultrasonic time of flight methods. Preliminary work has been conducted in a 5 laboratory EC/CERANORM project. Several key technical issues are unresolved with these methods. The VAMAS project could take several forms. It could be a conventional round robin wherein all participants conduct the same experiments, or the project could be a series of individual investigations by different laboratories on specific issues, or the project could be a combination of a baseline comparison round robin with individual laboratories free to investigate particular issues. Rectangular flexure type specimens as well as some disks would be available.

Status of Project: Discussion phase. Project to be developed and defined. A questionnaire about participants' laboratory capabilities and interests may be sent at a later date

5. *Wear resistance*

Dr. J-P. Erauw and Dr. Philippe Descamps of the Belgian Ceramic Research Center in Mons have proposed a new project for wear resistance evaluation. Jean-Pierre made a presentation that showed that CRIBC has a new model for correlation of wear resistance to basic mechanical properties such as elastic modulus, hardness, and fracture toughness. The new model differs from the traditional models in the literature. It different hardness and fracture toughness dependencies and includes elastic modulus:

$$W = K_{Ic}^{-2.19} HV^{0.8} \left[\frac{E}{HV} \right]^{.23}$$

The new model has been successfully applied to 90° particle erosion data (volume removal rates versus parameter W) for over one hundred materials! The proposal is to conduct a round robin on wear resistance of a range of ceramic materials. The specific test method is open for the time being. Testing could lead to development of standard test methods as well as furnishing data to interpret the various wear equations.

Mr. Quinn notes that such a project should be coordinated with VAMAS TWA #1, Wear. Mr. Quinn informed Dr. Santner at BAM and Dr. Hsu at NIST about this proposal.

Status of Project: Discussion phase. Project to be developed and defined.

OTHER

There has been no progress in developing the ***biaxial strength of aluminum nitride*** substrates project and this project has been dropped from further consideration.

Interactions with Technical Working Area 24, Electroceramics

Mr. Quinn has been in contact with Dr. Cain regarding the new TWA. The TWA 3 national leaders agreed that a new TWA was appropriate. Mr. Quinn met with Dr. Cain in London in June 1999 and agreed agree to coordinate their respective TWA's. Dr. Cain came to the Brighton TWA 3 meeting and spoke to the members about his plans. Dr. Mizuno then proposed four possible projects for TWA 24.

The TWA 3 web is accessible from the NIST Ceramic Division home page. The site includes a single page summary of the first 12 TWA #3 projects as well as general descriptions of TWA 3 and VAMAS. The address is: www.ceramics.nist.gov/webbok/vamas/vamastwa3.htm

1999 TWA 3 Publications:

37. J. Kübler, "Fracture Toughness of Ceramics Using the SEVNB Method: First Results of a Joint VAMAS/ESIS Round Robin," Ceramic Engineering & Science Proceedings, Vol. 20, Issue 3, 199, pp 495-502.
38. M. Mizuno, Y. Nagano, J. Kon, H. Tanaka and S. Sakaguchi, "Round Robin on Fracture Toughness Measurement of SiC-Whisker/Si_3N_4," presented at PACRIM 3, Kyongju, Korea, Sept. 1998.
39. C. Ullner and G. Quinn, "Interlaboratory Study on Depth Sensing Hardness on Ceramics," presented at IMEKO, Osaka, June 13-18, 1999.
40. J. Kübler, "Round Robin on Fracture Toughness of Ceramics Using the SEVNB Method (Single-Edge-V-Notched Beam)," VAMAS Final Report #37, EMPA, Dubendorf, Switzerland, 1999.
41. G. Quinn, "VAMAS After Twelve," Am. Ceram. Soc. Bull., Vol. 78, No. 7 July 1999 pp 78-83.
42. J. Kübler, "Fracture Toughness of Ceramics Using the SEVNB Method; Round Robin," VAMAS Report No 37 (also ESIS Report No.), 1999.
43. J. Kübler, "Fracture Toughness of Ceramics Using the SEVNB Method; Round Robin," A Joint VAMAS/ESIS Round Robin," to be publ. in proceedings of the Fracture Mechanics of Ceramics Conference, Moscow, July 1999.

VAMAS ANNUAL REPORT - 1999

TWA 5 - POLYMER COMPOSITES

International Chair: Graham D Sims, National Physical Laboratory

Date of report: April 2000

2. TWA 5 OFFICERS and PROJECT LEADERS

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PROJECT 1. - Assessment and Recommendation to ISO on Mode II

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PROJECT 2. - Measurement of Mechanical Properties for the Fibre-Matrix Interface

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PROJECT 3. - Measurement of Through-Thickness Properties

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PROJECT 4. - Damage Tolerance Assessment of PMCs

NPL as new leader c/o Dr Graham D Sims

3. PREVIOUS YEAR OVERVIEW

Meetings.

VAMAS international meeting held at the 12th International Conference on Composite Materials, Paris, July 1999, when suggestions for new areas were discussed. Next meetings planned at European Conference on Composite Materials, Brighton, July 2000 and Composites Testing and Standardisation : 5 in RISO, Denmark, 2001.

Publications.

"Comparison of test configurations for the determination of Mode 2 GII, - an international round robin" P Davies et al. Plastics, Composites and Rubber, November, 1999. VAMAS also mentioned in Guest editorial of this special issue on CTS:4 which included a special VAMAS session.

Standardisation

Based on the Mode II RR it was recommended and agreed at the ISO TC61 meeting in Sept. 1999 that "4-point ENF" be the preferred method for standardisation. Difficulties due to AFNOR requirement on funding of individual items suggest that it will stay at standstill until, AFNORs wider financial problems are resolved. Further input from the VAMAS round robin into ISO 13003 is being made as it progresses through standardisation. It is particularly disappointing considering that firstly, the project was organised to help ISO TC61/SC13/WG16 overcome a Japanese veto on Mode I progression without Mode II. And secondly, the effort, time and money expended by Dr Davies (France), the other participants and the material suppliers (Bell Helicopters and Toho) over two years that progress is now blocked due to the unique need of AFNOR to be paid for each standard development. As noted in the minutes abstracted below.-

ISO TC61/SC13 Minutes of 29/9/99 state regarding any new work item proposal on the agreed 4-point ENF Mode II method,

"The support from the industrial sector is absolutely necessary as well as supplemental support for the SC13 Secretariat The item will remain on idle until these conditions are met"

Dissemination

VAMAS mentioned in four other formal presentations* during the last year and on company visits (BAE SYSTEMS, ACG, DML). Need to assess fully dissemination outside the UK.

*

Pultrusion Conference - April 1999 Paris,
Aerospace conference Sept 1999 Toulouse,
Nottingham University Composites Club,
Thermal Methods Group- Royal Institution of Chemistry, Nov.1999
BRE Composites in Construction - three day conference Nov. 1999 and subsequent -day Network workshop, Jan 2000.

4. STATUS OF CURRENT PROJECTS:

PROJECT 1. - Assessment and Recommendation to ISO on Mode II (Completed)

Leader

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Project objectives

To assess several competing Mode II test methods in order to make a recommendation to ISO, thereby over-coming the Japanese veto on Mode 1.

Progress since last report

Second Round Robin (RR) completed. This allowed the recommendation on 4-pt ENF method to be made to ISO

Standardization activities

Based on the Mode II RR it was recommended and agreed at the ISO TC61 meeting in Sept. 1999 that "4-point ENF" be the preferred method for standardisation. Difficulties due to AFNOR requirement on funding of individual items suggest that it will stay at standstill until, AFNORs wider financial problems are resolved.

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"The support from the industrial sector is absolutely necessary as well as supplemental support for the SC13 Secretariat The item will remain on idle until these conditions are met"

Dissemination

"Comparison of test configurations for the determination of Mode 2 GII, - an international round robin" P Davies et al. Plastics, Composites and Rubber, November, 1999. VAMAS also

mentioned in Guest editorial of this special issue on CTS:4 which included a special VAMAS session.

PROJECT 2. Measurement of mechanical properties for the fibre-matrix interface

Leader

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Objectives

The program seeks to refine micro-mechanics test methods for assessing the fiber-matrix interface strength in composites, beginning with the best-known method, single fiber fragmentation. There are 3 specific objectives: (1) establish an accepted test protocol, (2) conduct a round robin to demonstrate that this protocol enables laboratories throughout the world to get equivalent results, and (3) generate an extensive database of information on one or more model systems so researchers can use this information to explore different data analysis models.

There is no current standard for interface strength testing although there are a variety of test methods that have been used to determine this parameter. The existing methods have been most successful for relative measurements as opposed to absolute determinations. Consequently, they are most useful for quality control and materials/surface treatment selection. The tests are used most widely by fiber companies and the aerospace industry. Although many other companies are interested in the tests, lack of standardization is a major barrier. There are two critical needs. First, it must be demonstrated that a standardized test procedure can give reproducible results in different laboratories. Second, the analysis procedure to calculate interface strength from the measured results must be improved to deal with realistic materials. For example, the current analysis methods assume elastic or elastic-plastic properties for the resin, but we know that the resins are non-linear viscoelastic. The work in this program addresses the first need directly and develops an extensive database of information on model systems as a first step toward addressing the second need.

Progress

At this time, the first three steps have been completed for the first model system. A decision was made to use two model systems: one ideally suited for the round robin and a second amenable to the Raman studies. Tests with the second model system will not be started until the round robin on the first is well underway. Before beginning the round robin, we must be sure that the samples give good results for a single laboratory. These preliminary tests should be complete by the end of July 2000. At that point, the round robin on the first model system (step 4) will begin. The round robin is expected to require 9 months. An additional 3 months is allocated to completion of other tests on the first model system (step 5). The analysis of results from the round robin is given an additional 3 months (step 6) so the target date for completion of the program for the first model system is the end of October 2000. Round robin tests with the second model system (step 4) should begin in January 2001 and are expected to take only 6 months. Allowing 3 months for steps 5 and 6 with the second model system means that completion of the program with the single fiber fragmentation tests and both model systems is scheduled for the end of July 2001.

International Participation and Industrial Interest

The list of participants is given below. Note that some laboratories are not participating with model system one. Although many industries are interested in interface testing (specifically, fiber makers, those involved in fiber surface treatments, and users who want to optimize materials for their application), most of these organizations contract the work to universities. The company most involved in the VAMAS study is Hexcel who has donated fiber for use in the program.

Dissemination

The major route for dissemination of results is publications and presentations at scientific meetings. In addition, the detailed information in the database will be distributed to anyone who is interested in studying the single fiber fragmentation test and its data analysis. Dissemination at USA meeting in June.

PROJECT 3. Measurement of Through-Thickness Testing Properties

Leader

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Objectives

- to review available techniques
- to develop new techniques
- to validate through-thickness test methods
- to obtain precision data in support of an agreed draft method (ISO NWIs?)
- to generate a database of through-thickness properties
- to obtain experience of these methods by an extended group of users.

The development of composite materials for structural applications in new sectors such as the ground transportation industry requires the use of thicker structures made of cost-effective materials (thick glass-epoxy woven composites, for instance). However, through-thickness properties become necessary to design such structures and only few and badly known methods are available. Therefore, there is an important need from industry to have such through-thickness test methods available. NPL has submitted some new drafts to BSI for ISO standards and ASTM has some related items.

Progress

Prof. Pierron has just changed posts and has not been able to initiate work yet. The associated EU Framework 5 network proposal was submitted in March 2000.

Dissemination

Planned to be via International conferences (e.g. ICCM/12, CTS/5, ECCM-9), standards committees (ISO TC61/SC13/WG16 and CEN TC 249/SC2/WG5 as draft methods and/or precision data in support of such drafts) and via EU Framework 5 network.

PROJECT 4. ASSESSMENT OF DAMAGE TOLERANCE FOR PMCs

Leader

Proposal to be re-formulated following change in project leadership to NPL.

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5. NEW PROPOSALS

Other topics under consideration (no formal proposal)

Potential topics include:

- Thermal analysis techniques (DSC, DMA), based on enquiries received from other countries (e.g. Italy, USA) to join UK Studio Project.
- Structural element tests, such as open hole compression and tension, and pin bearing tests. These tests cover more than just aerospace interests do. Some intercomparison of NPL proposals and ASTM preferences, as well as the wider generation of precision data to supplement an UK RR.
- Ultrasonics for: a. defect detection during NDE inspection,
  b. measurement of elastic constants.
- Composite adhesion tests (peel-static/dynamic, weathering, edge delamination (fatigue), heat cycle) related to: a. composite bonded repair of structures,
  b. composite stiffened conventional materials,
  (Proposed under IEA/OECD Climate Initiative (CTI) by Japan).
- Collaboration on design data database formats based on the technical data sheet standard, ISO 10350-2, drafted at NPL.
- Collaborative international long-term durability studies to generate a larger database.
- Processing properties test methods.

Survey to be undertaken of national co-ordinators to determine priorities for future programmes.

6. FUTURE NATIONAL/REGIONAL/INTERNATIONAL MEETINGS

Next meetings planned are at:

- European Conference on Composite Materials, Brighton, July 2000
- Composites Testing and Standardisation : 5 in RISO , Denmark, 2001,
- International Conference on Composite Materials, Bejing, 2001

TWA 7 - BIOMATERIALS

ANNUAL REPORT

T. Tateishi

May 2, 2000

Evaluation of cytotoxicity of UHMWPE wear debris

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Introduction

Osteolysis and subsequent loosening of implants are considered to be most significant problem in total joint replacement. Many reports suggest that retrieved periprosthetic tissues contain significant volumes of ultrs-high-molecular-weight polyethylene (UHMWPE) particles [1-3]. This also consistent with the view that UHMWPE wear debris plays an important role in osteolysis. In particular, polyethylene particles activate macrophages which stimulate osteclasts, leading to bone resorption and loosening. In spite of the fact that UHMWPE is one of the major constituents of wear debris, very few have investigated the phagocytosis of polyethylene particles by macrophage *in vitro* [4-7]. This may be due to the technical difficulties in handling polyethylene particles as they have a reletively low density than water and tend to float at the surface of the aqueous medium rather tha to be at the bottoom of the tisuue culture wells. The authors in this work have used an inverted cell culture method [8] to study the phagocytosis of the polyethylene particles obtained by both in vivo and in vitro methods using murine mouse macrophage by primary cell culture method. The five different samples of polyethylene of varied size used in this work were from (1) Pin on plate wear test machine (2) *In vivo* samples 1 and 2 and (3) knee and the hip wear simulator. Also for comparison commercially available polyethylene beads were investigated.

Cytotoxicity of wear debris

Cytotoxicity index of wear debris were evaluated by measuring the flourescence with Alamar Blue assay to asses the activity of the cells and and optical density measurments with Nuetral Red assay to assess the number of cells. The particle size distribution of the different wear debris and polyethylene beads used in this work were performed by analyzing the images of the SEM photographs by using image analysis software (NIH image). The mean diameters of polyethylene beads, pin on disk wear debris, *in vivo* wear debris, Knee simulator wear debris and hip simulator wear debris were 3.43, 1. 53, 0.25, 0.51 and 0.53 micrometers respectively. The cytotoxicity index is the ratio of Flourescence (AB assay) or OD (NR assay) of wear debris to the Flourescence (AB assay) or OD (NR assay) of control (without wear debris).

Objective

The initial objective of this work was to study the phagocytosis of wear debris obtained from different sources with varying size. So we used polyethylene particles from different sources, and also their mean diameters were estimated from SEM photographs. A correlation between size and activity of cells and the number of cells were made. At present the various cytokine release like IL-1, IL-6. TNF-α are being under investigation. Later we would like to investigate iNOS messenger RNA and our final objective is to is to deduce the signal transdution pathways which ultimately leads to bone resorption and thus resulting in loosening of the implant.

Work plan

We would like to carry out this work in three phases.

1. In the first phase, we tried to correlate the wear particle size obtained from different sources with activity and the number of cells using Alamar Blue and Nuetral Red assays. Also, SEM analysis were carried out to understand the morphology of the wear debris from which we calculated the mean diameters with an image analysis software (NIH image).
2. In the second phase we would like to concentrate on the various cytokines released by the macrophage cells on being challenged with wear particles obtained from different sources.
3. In the third and the final phase, we would like to evaluate the signal transduction pathways which ultimately leads to bone resorption resulting in the loosening of the implant.

Evaluation method

We developed an inverted cell culture technique (Fig.1) to study the phagocytosis of the polyethylene wear debris using primary macrophage cells. The activity and the number of the cells after phagocytosis process were estimated by Alamar Blue and Nuetral Red assay techniques. Variuos cytokine release would be studied by ELISA and other techniques. We are currently using RT-PCR, in-situ hybridization western blot analysis to study the gene expression and protien analysis.

Results

Figure 2 shows the results of Alamar Blue and Neytral Red assays for polyethylene beads, debris from pin on disk machine, from *in vivo* samples 1 and 2 and debris from the knee and the hip simulator. The assay result for polyethylene beads and from the wear debris from the pin on disk were almost equivalent as that of control. The activity and the number of cells do not vary much from the control, while for the wear debris from the *in vivo* experiments, there was a significant difference from the control, PE beads and the debris from the wear test machine.

Figure 3(a-e) shows the SEM photographs of polyethylene beads, pin on disk wear debris, debris from *in vivo* sample, wear debris from the knee and hip simulator, respectively. It can be seen that the size and shape of the particles widely varied for the different UHMWPE wear debris obtained from different techniques. Moreover, the wear debris from the *in vivo* samples were more spherical in shape than from the wear debris obtained from other techniques. This may be due to the fact that the wear debris from the *in vivo* samples were collected after 5 years of implantation at the knee and thus particles ultimately becomes spherical in shape due to the constant movement of the knee. On the other hand, the wear debris from the knee and the hip simulator had no definite shape and were scattered too.

Figure 3(f) shows the optical micrographs of phagocytized UHMWPE particlesby macrophages. The “arrow” indicates the UHMWPE wear particles. The size distribution of polyethylene beads and the variuos UHMWPE wear debris is shown in Fig. 4. More than 30% of the in vivo wear debris as shown in the fig 4(c) had diameters between 0.4 to 0.6 micrometers and this was the smallest mean diameter as compared to that of any other wear debris investigated in this work. Diameter as calculated from area of the particles decreased in the order polyethylene beads>pin on disk wear debris>hip simulator wear debris>knee simulator wear debris>*in vivo* wear debris. On the contrary, the cytotoxicity the cytotoxicity index decreased in the order *in vivo* wear debris <knee simulator wear debris<hip simulator wear debris<pin on disk wear debris<polyethylene beads. Hence, it could be presumed that the particles with a smaller size were phagocytised more effectively than those with higher size.

To make a comparison with activity of the cells and the number of the cells, the mean particle diameters were plotted against the cytotoxicity index for both Alamar Blue and Nuetral Red assays as shown in Fig. 5(a,b). It could be seen both the activity and the number of cells were low for the *in vivo* wear debris sample, thus indicating that phagocytosis process has a relationship with the particle size. It can also be seen that the activity of cells sharply falls for the wear debris from the knee simulator, whereas there was not much effect on the number of cells for the wear debris from the hip simulator. From the present data it could be concluded that after 4 h of exposure of UHMWPE obtained from diffarent sources the cytotoxicity is more for particles with smaller size as compared to that of polyethylene beads with a mean diameter of 3.43 micrometer.

Conclusions

From the SEM photographs and the subsequent image analysis it was found that *in vivo* wear debris had the smallest diameter as compared to other UHMWPE wear debris investigated in this work. Also, cytotoxicity index were significantly low for *in vivo* wear debris. Inverted cell culture method may be considered as one of good methods to study the phagocytosis of UHMWPE wear debris.

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TWA 10 Annual Report

1999

Computerized Materials Data

Dr. Kohemi Halada

TWA 10 (Computerized materials property data) has two activities; one is STEP TERMINOLOGY REVIEW and the other is GENERIC DATA-SHARING PLATFORM. The project in Step Terminology Review, Project No. 1, started in 1998 with the project name **"Review of STEP Materials Terminology"** proposed by Dr. J. Carpenter, USA. The objective of this project is to ensure consistency in definitions of entities and attributes among various STEP application protocols used for trhe computerized exchange of information about materials. Project No. 2, **"Investigation on the generic platform for electrical data sharing system of materials data",** under the Generic Data-sharing Platform, was initiated in 1999 by Dr. K. Halada, JPN,. The objective of this activity is to clarify the prerequisite for the generic platform for electrical data-sharing systems of materials data. In order to promote the data-sharing system from multi-sources of materials data, where each database has its own inherent structure, it is necessary to prepare a common basis to retrieve, refer, link and utilize the data among them with electrical exchange.

The background of pre-standardization, plans and the current status of these activities are as follows;

STEP TERMINOLOGY REVIEW

STEP is an unofficial acronym (Standard for the Exchange of Product Model Data) for ISO 10303 for the computer-to-computer exchange of technical information on manufactured products throughout their entire life cycles. The first STEP standards, issued as formal ISO International Standards (IS) in December 1994, primarily dealt with the fundamental concepts within STEP and its use in computer-aided design (CAD). Other STEP standards are nearing IS status that will fulfill the original intent of covering all data on products throughout their entire life cycle, not just the design phase. STEP is intended to facilitate enterprise integration by simplifying computer systems integration no matter what types of computers or software are used or what natural languages (tongues) are spoken by the persons in the organizations involved in the enterprise. Fundamentally, the standard is for exchange of *unequivocal* concepts taken bite-wise and in a relatively well confined context (e.g., a specific industry or technical field) for some specific activity in that context (e.g., engineering or scientific analyses). STEP does this by providing internationally agreed-to (consensus) *meaning* and *structure* to the information when exchanged among computer systems. This is done by means of a standard called an application protocol (AP) which is basically a written set of explicit rules ("protocol" in STEP-ese). The application protocol stipulates that when one wants to exchange technical information for some activity ("application" in STEP parlance) between computers, the information must be sequenced in a specified way and the concepts being exchanged must be clearly defined and understood (unequivocal) by all parties involved. The intent of the VAMAS effort is to minimize as much as possible during the exchange the use of technical-field-specific terms for identical concepts, thus promoting cross-fertilization between technical fields and reusability of APs for different activities.

As part of this project, a Review Board is being established. The Review Board is planned to consist of two boards; one is a *coordinator's board* and the other is an *expert's board*. The members of the coordinator's board are not necessary to be experts in the subject field in the AP. The coordinator's board identifies the experts well suited for the subject AP for the expert's board. The chief of the coordinator's board is Dr. Carpenter, and members from the U.S.A., Japan and Italy have been determined.

The first AP is AP 223 (Aluminum Foundry). As soon as the draft of AP 223 is prepared, the expert's board will be organized to review the terms of materials associated with Aluminum foundry. Other fields associated with materials data can be treated as the same manner. As LCA (Life-Cycle Assessment) data is a possible subject for this activity, collaboration between the group concerned with STEP and that with LCA is the subject of negotiation at this time. The review board will be able to start as soon as the AP for LCA is prepared.

GENERIC DATA-SHARING PLATFORM

The necessity of computerized data exchange of materials data is increasing, especially in the field of advanced materials for the agile exchange of information, because databases of materials data are widely distributed all over the world. However, a common procedure to retrieve and use the data from the

distributed database does not yet exist because each database has its own format and structure. The guideline and standardization of individual databases have been made in ISO 9075 (Database language), ISO 9579 (Remote database access), and ASTM E49 especially for materials data. Further development of common or standardized procedures for the data exchange system from the viewpoint of the common platform on which data can be treated without regard to the structure of the original database, is required in today's computerized era.

For this purpose, the standardized "generic platform" and the "standardized template", through which each existing database can be put on the platform without changing its inherent structure should be prepared. The generic platform and the standardized template will accelerate the data exchange in the following ways;

- A database distributed individually can be treated as a part of a universal database on the generic platform when the database adheres to the "standardized template."

- Personal databases developed by researchers or investigators can be easily collected by communicating with the standardized template. This will enhance the development of the designing of various advanced materials.

- Various properties of materials stored in different databases can be linked on the generic platform with the standardized template, in order to effect life-cycle design of products from comprehensive approaches such as DfE (Design for Environment) DfS (Design for Safety), etc., used by industry.

The plan will be carried out in three stages.

Stage I: Feasibility study on the electrical data-sharing platform of distributed materials data

- comparison of the structure among various materials databases
- review of standardized or pre-standardized meta-data structure for materials data
- review and comparison of the descriptions of data structure definition in XML and EXPRESS or other possible data description languages
- extraction of requisites for electrical data-sharing among distributed materials data

Stage II: Trial and testing the prototype of DTD template for existing database

- creation of prototype Document Type Definition (DTD) for several existing databases
- documentation of DTD from pre-standardized database structure such as specified by ASTM E49 standards
- comparison and testing with retrieval
- clarification of the requisite of the generic DTD structure for material data

Stage III: Establishment of the common template and establishment of the common data-sharing platform

- decision of generic DTD for the common template for materials data
- proposal of the data browsing, retrieval and linking system on the common platform

Stage I (feasibility study) began in 1999. Two research efforts have started in Japan and in U.S.A. The New Materials Center in Japan organized the XML-DTD Group, conducted by Prof. Horikawa, and investigates the common DTD description for materials property database. XML-DTD group is not focused on the common format of database, nor on the common meta-data format/structure, but on the customizable description of meta-data structure by XML. The framework of the materials database in ASTM E49 standards and the structure of existing databases, such as fatigue, creep, sintered metals, heat resistant metals, amorphous, etc., were considered as a referential DTD for materials database. The first draft of the referential DTD is published on the page of "projects" in the homepage of TWA10

(http://www.nrim.go.jp8080/vamas_twa10/). The format for the environmental load data of materials for LCA, now being discussed in ISO TC207/SC5, is also being considered by this group. The example of DTD for LCA data is also listed in the same homepage.

Dr. E. Begley, NIST (USA), leads an effort to develop MatML (materials markup language) and is investigating the fundamental basis of MatML. The MatML group opened a discussion page in the homepage of NIST and it is used mainly as a mailing list for the discussion. The scope of MatML, the application of MatML, the necessity of special software, the extensibility of MatML, and the leverage of other specifications were also put on the discussion page as discussion points. As the result of the investigation, version 1.0 of MatML has been released; it consists of "data source", "material", "property data", "term", and "graph." The MatML version 1.0 is also published on the NIST web site. The URL is http://www.ceramics.nist.gov/matml/matml.htm.

The achievement of these efforts to develop generic data-sharing platform have been taken into account in the planning of the project as well as the increased possibility of linkage with an expanded STEP as an application of MatML. This progress necessitates TWA 10 to clarify the practical target and to concentrate collaboration work in the Stage II. The interchange of these efforts will be done in the coming CODATA conference in this October. A key persons meeting will be held in Japan after the CODATA conference in order to clarify and modify the practical plan of Stage II. Stage II will start from 2001 reflecting the result of key persons meeting.

Action plan and request for Steering Committee action

For STEP TERMINOLOGY REVIEW

10-1. VAMAS SC appoints Dr. J. Carpenter as the co-chair of TWA 10 associated with STEP TERMINOLOGY REVIEW project.

10.2. TWA 10 moves quickly to identify members of Coordinator's Board. VAMAS SC extends invitation to join the Coordinator's Board to those countries whose coordinator member has not been determined.

10.3. TWA10 assists to accelerate the documentation of AS223 (Aluminum Foundry) of STEP.

10.4. TWA10 asks Coordinator's Board to immediately start the review by the expert's board when the AP document is prepared.

For GENERIC DATA-SHARING PLATFORM

10.5. TWA 10 postpones the switchover this year to Stage II from the feasibility study stage in order to modify the action plan of Stage II.

10.6. TWA 10 modifies the time schedule, testing methods and participants of the GENERIC DATA-SHARING PLATFORM effort to consider the progress in USA (MatML) and Japan (XML-DTD for materials database) and to consider the collaboration with STEP.

10.7. TWA 10 holds a key-persons meeting in Japan in late 2000 winter, in order to clarify the practical action plan in Stage II and III.

ANNUAL REPORT

VAMAS Technical Working Area 13

LOW CYCLE FATIGUE

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April 2000

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Overview of Activities Since Last Report

The final report on the VAMAS/EC project on "*Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces'* was produced in October 1999. Dissemination of outputs from the work has continued through publications, oral presentations at public meeting and contributions to national and international standards bodies. The findings from the work are making significant impact onto the development of at least 6 new standards and best test practice in this area.

Status of Current Projects

Project title: Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces

Project leader: Dr F A Kandil, National Physical Laboratory

Project Objectives:
(1)To develop a framework for quantifying measurement uncertainties in LCF data
(2)To validate the new Code of Practice for the measurement of bending caused by load misalignment in uniaxial fatigue testing

(3) To provide recommendations for a best practice for routine LCF testing of metallic materials.

Progress since last report: Work completed.

Standardisation and dissemination activities:
NPL and NRIM contribute to the work of the ISO/TC164/SC5 Committee on Fatigue. This Committee meets annually and the last meeting was held at AFNOR, Paris, 5-6 October 1999.

F A Kandil, "Potential Ambiguity in the Determination of the Plastic Strain Range Component in LCF Testing", Presentation at a workshop on 'Cyclic Stress-Strain Data Testing and Interpretation', High Temperature Testing Committee (ESIS TC11), Institute of Materials, London, 7 December 1999.

F A Kandil, "A Method for the Measurement of Alignment of Uniaxial Test Systems", Presentation at the 'National Measurement Conference 99', Brighton, UK, 2-4 November, 1999.

F A Kandil, "Quantifying Data Uncertainties and the Validation of a Code of Practice for the Measurement of Bending in Uniaxial Fatigue Test Pieces", Final Report, Project MAT1-CT94-0079, NPL, October 1999.

F A Kandil, "Potential Ambiguity in the Determination of the Plastic Strain Range Component in LCF Testing", Int. J. Fatigue, Vol. 21, No.10, November 1999, 1013-18.

Proposed Future Projects

In the last report it was mentioned that a new project on evaluation of residual stress measurement techniques (with specific emphasis on the XRD and hole drilling methods) started at NPL in April 1999. A number of organisations including JRC Petten, expressed an interest in this work. Plans are in hand to circulate information on the current work objectives and plans with the view to formulate a complementary research programme on effects of residual stresses on fatigue behaviour.

Future National and International Meetings

"Fatigue 2000", 4th International Conference of the Engineering Integrity Society, Robinson College, Cambridge, UK, 10-12 April 2000.

EuroMat 2000, Tours, France, 7-9 November 2000.

Metal Matrix Composites

Chitoshi Masuda

National Research Institute for Metals

April 27, 2000

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General Overview of activities:

Develop understanding of mechanical property determination on tensile tests and fatigue tests of metal matrix composites having heterogeneous structures at room and elevated temperatures and establish a unified and reliable testing method through a series of Round Robin Tests (RRT).

Status of Current Projects

1) SiCw/A2009 MMC

Last VAMAS TWA 15 meeting was held in Paris, France on July, 1999. The preliminary data from fatigue tests at room temperature for SiCw/A2009 Metal Matrix Composites (MMC) obtained at NRIM were discussed. Prof. W. S. Johnson (Georgia Tech) pointed out that 500 MPa should be selected. The slope of the curve on the stress-number of cycles to failure relationship was not so steep. If the data were obtained at the lower stress level according to the test conditions authorized at the TWA 15 meeting (the stress level should be selected at the number of cycles to failure of about $5x10^5$ cycles), the scatter band of data would be larger. For example, if the failure life would be more than 10^7 cycles, the test method could not be discussed.

Moreover Prof. Johnson proposed that the specimen surface and edges should be polished and the edge corners should be machined round (0.5c) to reduce the fatigue crack initiation sites. After the data for the polished specimens were obtained at NRIM, Prof. Johnson checked the data and macroscopic fracture surfaces of the fatigue specimens and he agreed with the test specimen conditions. After the test specimens were machined and polished at NRIM, they were sent to NPL (UK). Dr. J. Lord then distributed them to the participants of TWA 15. The fatigue testing at 200 deg. C was performed to obtain the preliminary data at NRIM.

2) SiC/Ti-15-3 MMC

The preliminary fatigue test data for small-width specimens (2.5 mm in width) obtained at NRIM were discussed. Prof. Johnson pointed out that the data for wide-width specimens (about 100 mm in width) should be referred for RRT to assess the test stress condition because the fracture mechanism for small specimens was different from that for large specimens. Therefore the wide-width specimens should be tested in order to obtain the preliminary data. NPL machined the specimens of 10 mm width and Prof. Johnson agreed to test them. The fatigue test was carried out at 480 deg. C at the Georgia Institute of Technology (USA). The specimens have already been machined at NPL and they would be distributed after obtaining the preliminary data.

3) Al_2O_3/Al MMC

The materials have already been sent to NPL from 3M (USA) and the nondestructive testing results of all materials using ultrasonic technique(C san) had also been received (NPL) from Dayton University (USA). Prof. Johnson agreed to ask 3M for the preliminary tensile and fatigue test data of this material for tests at room temperature and at 200C. If the preliminary data information can not be obtained, tensile and

fatigue tests should be performed in order to obtain the preliminary data. Unfortunately the information has not been received yet.

Standardization activities

1) ISO/TTA2,"Tensile test for discontinuous fiber reinforced metal matrix composite at ambient temperature",(1998)

2) VAMAS Report No.23, "Elevated temperature tensile tests on discontinuously reinforced MMC-at approval stage",(1996)

3) VAMAS Report No.27, "Elevated temperature tensile tests on discontinuously reinforced MMC", (1997)

4) VAMAS Report No. 30,"Tensile test data at elevated temperature on discontinuous reinforced MMC", (2000)

Propose future Projects:

The questionnaire has already been sent back to NPL from the members of TWA 15 and other researchers who engage in the development and evaluation research of metal matrix composites in advanced countries. Dr. J. Lord has analyzed many items on the questionnaire. The next projects would be proposed after discussion of the report of the questionnaire.

TWA ANNUAL REPORT

TWA-16 Superconducting Materials

Chairperson: H. Wada, National Research Institute for Metals, Japan

April 30, 2000

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Status of Current Projects

(1) Project number: WG1-1
Title: Bending strain effects on critical current in oxide superconductors

Project leader: T.Kuroda, National Research Institute for Metals

Project objectives: To establish a standard measurement method on the critical current of Bi-2212/-2223 oxide superconductors as a function of bending strain. Critical currents of Bi-2212/-2223 oxide superconductor specimens are measured at 77 K and with no external field, after bending each of the specimens to a different, designated curvature at room temperature.

Progress since last report: Preliminary measurements revealed that critical current strongly depends on how to bend the specimen. To avoid ambiguity in the bending procedure, specially designed holders were used, each consisting of a G-10 substrate with a curved surface and two current contacts. The measurement procedure was designed in detail for a worldwide round robin test (RRT). Three kinds of Bi-2223 Ag and Ag-alloy sheathed multi-filamentary tapes have been prepared as RRT specimens.

Standardization activities: A standard draft will be proposed to IEC after the RRTs are completed and discussed by the TWA members.

Talks/Reports/Publications based on project results:
None

(2) Project number: WG1-2
Title: Measurement method of critical temperature in oxide superconductors

Project leader: Y.Kimura, Ehime University

Project objectives: To establish a standard measurement method on the critical temperature of oxide superconductors.

Progress since last report: Comparisons among resistive, dc and ac magnetization methods were performed among participants through RRTs. It was then concluded that the resistive method is recommendable as a standard method for measuring critical temperatures of oxide superconductors.

Standardization activities: The first draft of a recommended standard was prepared in November 1999 and discussed by TWA 16 members. An IEC standard draft was prepared and proposed at the IEC/TC90 committee meeting at NIST, Boulder in March 2000.

Talks/Reports/Publications based on project results:
None

(3) Project number: WG2-1
Title: Measurement methods for trapped field and levitation force in bulk oxide superconductors

Project leader: M.Murakami, Superconductivity Research Laboratory

Project objectives: To establish standard measurement methods on trapped flux density and levitation force of bulk superconductors. The trapped flux density profile of a bulk high-Tc oxide superconductor is measured by scanning a Hall probe on the surface of a specimen. The magnetization process in a strong magnetic field during cool-down performs the flux trapping.

Progress since last report: Preliminary studies were made on trapped flux density and levitation force measurement methods. In the trapped flux density measurement, specimen breakdown occurred during the magnetization process. This problem has now been solved by finding that impregnation of some kinds of resin is effective to prevent the sample from cracking. In the levitation force measurement, it has been found that the force is approximately proportional to the base area of the bulk superconductor.

Standardization activities: An IEC standard draft for the measurement of trapped flux density was prepared after the discussions by TWA 16 members and proposed at the IEC/TC 90 committee meeting at NIST, Boulder in March 2000.

Talks/Reports/Publications based on project results:

1) M.Tomita and M.Murakami; Improvement of the mechanical properties of bulk superconductors by the impregnation of epoxy resins, Teion-Kogaku (Japanese), 34 (1999) 617-20

(4) Project number: WG3-1

Title: Measurement methods for the surface resistance in thin film superconductors

Project leader: S.Kosaka, Electrotechnical Laboratory

Project objectives: To confirm standard measurement method, IEC standard draft IEC 61788-7, on the surface resistance in thin film superconductors proposed at the 1998 IEC/TC 90 meeting in Frankfurt. The target accuracy for the measurement method proposed is within 10 %, which is now being examined through RRTs within TWA 16. The surface resistance is determined by applying microwave signal to a cylindrical dielectric resonator sandwiched with two superconducting thin films and measuring the insertion attenuation of the resonator at some frequency between 8 GHz to 30 GHz.

Progress since last report: A preliminary RRT was implemented in 1998-9 in Japan. The resulting data scatter was less than 10 % and small enough. The first RRT was implemented at the beginning of 2000. The resulting data scatter however became much larger. The major source of the data scatter was supposed to be inhomogeneity of the dielectric rods (sapphire) used in the measurements. The second RRT is now in action and will be completed in June 2000, in which a unique sapphire rod will be used to minimize the error from dielectric inhomogeneity.

Standardization activities: The IEC standard draft, IEC 61788-7 is now on CD stage. It will move to the CDV stage by refining the draft based on the results of the second RRT.

Talks/Reports/Publications based on project results:

1) Ohara et al; Cryostat for surface resistance measurement by dielectric resonator method, presented at 47th Oyobutsurigaku-kankei-rengokoenkai, 1999

(5) Project number: WG4-1

Title: Measurement method for the mechanical properties of oxide superconductors

Project leader: K.Osamura, Kyoto University

Project objectives: To establish standard measurement methods on mechanical properties of oxide superconductors, such as yield strength and Young's modulus. The measurements are performed at room temperature.

Progress since last report: A RRT is being implemented, where the measurement conditions are not specified at all. The participants are requested to measure by their own techniques. The RRT will be completed within this year. Another RRT will be implemented, where the measurement conditions are well specified.

Standardization activities: In preparation.

Talks/Reports/Publications based on project results:

1) K.Osamura et al; Definition of mechanical properties assessed by room temperature tensile test for the Cu/Nb-Ti composite wires, Adv. in Supercond., XI (1999) 1515-8

2) K.Osamura et al; Inhomogeneous transport properties in Ag/Bi2223 tapes, Physica C, 3614(2000)xxx

Proposed/Future Projects

1. Project number: WG1-3
   Title: Measurement method for the irreversibility field in oxide superconductors

   Project leader: T.Matsushita, Kyushu Institute of Technology

   General focus/objective: To clarify the relation between V-I characteristics and flux pinning, determine the relation among irreversibility (critical) fields obtained by different measurement methods and establish an adequate and reliable measurement method.

   Discussion of need: Irreversibility field or critical field is one of the critical parameters, beyond which superconductors cannot carry any effective superconducting current. This parameter is important for practical applications as well as in material characterization.

List of Future National/Regional/International meetings relevant to TWA activities

1. TWA 16 meeting, Tokyo, October 2000.
2. International Superconductivity Symposium (ISS 2000), Tokyo, October 2000.
3. Applied Superconductivity Conference (ASC 2000), Virginia Beach, VA, USA Sept. 2000

TWA 17

Cryogenic Structural Materials

Toshio Ogata

National Research Institute for Metals

April 23, 2000

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General overview of activities:

- The 14th Technical Working Party meeting was held on July 12, 1999, in Montreal, Canada. Results of three projects and activities for international standardization were discussed. Status of current projects is described below.
- A draft of New Work Item of ISO/TC164/SC1 titled “Metallic Materials - Tensile Testing in Liquid Helium” was submitted to the ISO/TC164/SC1 and was distributed to the SC1 members for voting.

Status of Current Projects

1) Project number and title: No. 5, Interlaminar Shear test on GFRP
Project leader and affiliation: Prof. Y. Shindo, Tohoku University

Project objectives:
Develop understanding of mechanical property determination on interlaminar shear tests of GFRP at liquid helium temperature and establish a unified and reliable testing method through a series of RRT.

Progress since last report:
Testing procedures with the advanced specimen geometry and specimens for the third round robin test were distributed to participants. The results were good as expected and the report was presented at the ICMC'99.

Standardization activities:
Agree to prepare a TTA documents as a technical report for ISO/TC 61.

Talks/Reports/Publications:
Y. Shindo, R. Wang, K. Horiguchi and S. Ueda, Theoretical and Experimental Evaluation of Double-Notch Shear Strength of G-10CR Glass-Cloth/Epoxy Laminates at Cryogenic Temperatures, ASME Journal of Engineering Materials and Technology(121, 3, 367-373, 1999)

2) Project number and title: No. 6, Mechanical tests in high magnetic field
Project leader and affiliation: Prof. K. Shibata, University of Tokyo

Project objectives:
Establish reliable methods of evaluating Young's modulus, yield strength, tensile strength, elongation, fracture toughness at 4K in high magnetic field.

Progress since last report:
Preliminary tests at 4K in High Magnetic field were carried out in Germany and Japan, and little effect of magnetic field on load-cell and/or extensometer was confirmed in proposed testing system. The specimens of titanium alloy for the first RRT have been distributed to the participants.

Standardization activities:
None at this time

Talks/Reports/Publications:
K. Shibata, T. Kadota, Y. Kohno, A. Nyilas, and T. Ogata,
MECHANICAL PROPERTIES OF A BORON ADDED SUPERALLOY AT 4K AND MAGNETIC EFFECT, International Cryogenic Materials Conference, Montreal, July, 1999

3) Project number and title: No. 7, Advanced fracture toughness test (J evaluation on tensile test by round bar with circumferential notch):
Project leader and affiliation: Prof. A Nishimura, National Institute for Fusion Science

Project objectives:
Develop a understanding and testing method of fracture toughness determination at liquid helium temperature and establish a testing method using a small-size specimen for the test through a series of RRT.

Progress since last report:
Testing procedures and specimens of the test for the second round robin test were distributed to participants. Five institutes have reported the results so far and tentative results were presented at the ICMC'99 last July.

Standardization activities:
None at this time

Talks/Reports/Publications:
A. Nishimura, T. Ogata, Y. Shindo, K. Shibata, A. Nyilas, R. P. Walsh, J. W. Chan, and H. Mitterbacher, Local Fracture Toughness Evaluation of 316LN Plate at Cryogenic Temperature, International Cryogenic Materials Conference in Montreal, July 12-16, 1999 IGA-1

A. Nishimura and T. Ogata, Local Fracture Toughness Evaluation by JETT at Cryogenic Temperature, 61th Meeting on Cryogenics and Superconductivity, Japan, November 10-12, 1999

3) Other standardization activities

Progress since last report:
A draft of New Work Item of ISO/TC164/SC1 titled “Metallic Materials - Tensile Testing in Liquid Helium” was submitted to the ISO/TC164/SC1 according to a request of the SC1 and the agreement of the last TWP meeting. The proposed draft was distributed to the SC1 members for voting.

Talks/Reports/Publications:
T. Ogata and Participants of VAMAS TWA 17, Results of VAMAS Activities on Pre-standardization of Mechanical Properties Evaluation at 4K, International Cryogenic Materials Conference, July 12-16, 1999, IEB-6

T. Ogata and T. Yuri, Results of VAMAS Activities of Standardization of Evaluation of Cryogenic Structural Materials – 3, 61th Meeting on Cryogenics and Superconductivity, Japan, November 10-12, 1999

Proposed/Future Projects:
none

List of Future National/Regional/International:
Meeting on Cryogenics and Superconductivity 2000 – Spring, May 29-31, 2000.
International Cryogenic Materials Conference, in Madison, USA, July 2001.

TWA 18 ANNUAL REPORT

1. TWA 18 ``Statistical Techniques for Interlaboratory Studies and Related Projects''

Chairman: Dr. Jörg Polzehl, Weierstrass-Institute of Applied Analysis and Stochastics
Berlin, Germany
Date: April 16, 2000

2. List of active TWA officers

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3. General Overview

Change of TWA Chairman from Mr. Thomas Fritz, BAM, Germany to Dr. Jörg Polzehl, WIAS Berlin, Germany. Main current task is the reorganization of the TWA as a body for statistical consulting and advice within VAMAS.

4. Current Projects

There are no current projects within TWA 18. The role of TWA 18 is to support the work of other TWA by offering advice and statistical consulting. In general it is not intended for the TWA to initiate own projects.

5. General Comments

Statistical reviewing of testing protocols is recommended in the ``VAMAS Project Initiation Form'' used when initiating a new work project. Members of the TWA or (and) statistician from coordinating institution should be involved into new projects already at the time of project initiation. To do so I'd like to request an information about new projects at the point of project initiation.A similar attempt has a already been made e.g. in 1996 (see VAMAS Bulletin #20 1996, pp. 17/18), but does not seem to work in the moment.

Responsibility for the statistical planning and analysis should in most cases be with statisticians from the organization initiating or coordination a project. I'd consider this necessary in order to secure the necessary close contact to the scientists responsible in the field of application. The TWA will serve as a body of consultancy for specific questions and problems. To do so we'll need to interest statisticians from institutions involved in VAMAS to participate in the work of the TWA. Currently we have no member from either NIST and NPL.

April 2000

VAMAS TWA 20

MEASUREMENT OF RESIDUAL STRESS

CHAIRMAN: Professor George A. Webster
Department of Mechanical Engineering
Imperial College
London, SW7 2BX, UK

23rd March 2000

2. Project Leader

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3. Overview

Currently TWA 20 is concerned with the measurement of residual stress by neutron diffraction. Four Round Robin samples are being examined. These include a shrink-fit aluminium alloy ring and plug assembly, a ceramic matrix composite, a shot-peened nickel alloy plate and a ferritic steel weldment. These samples were chosen to represent a range of materials and practical applications of industrial importance. Measurements are almost complete. During the past year progress meetings have been held at the JRC, Petten, Holland from 7-9 July 1999 and at LANSCE, Los Alamos, USA from 22-24 February 2000. As an innovation at this latter meeting, a one-day scientific pre-meeting was held prior to the main meeting to discuss new developments in the modelling, significance and measurement of residual stress. This proved to be extremely interesting and beneficial. Analysis of the results on the ring and plug assembly is complete and the findings have been issued in the following report:

VAMAS REPORT No 38 'Neutron diffraction measurements of residual stress in a shrink-fit ring and plug', (Ed. G.A. Webster), Jan 2000. Issued by NPL, UK

Interpretation of the measurements on the other samples is well in hand and is expected to be presented at the next progress meeting which is due to take place at ISIS, Abingdon, UK from 13-14 July 2000. By this time all measurements are anticipated to have been completed.

4. Status

4.1 Standardization Activities

Considerable progress has been made in assembling the necessary information for preparing a draft standard for making measurements of residual stress by neutron diffraction. A protocol has been agreed for obtaining reliable results. An outline document has been produced and a revised version will be prepared for discussion at the next meeting. When approved it is intended that it will be issued as an ISO/VAMAS TTA document prior to submission as the basis for a standard. Discussions have been held with ASTM and CEN concerning the preparation of a standard. It is evident that both routes are possible.

4.2 Publication of results

The results of the investigation have been distributed widely. A web site has been set-up to report progress. Its address is: http://www.risoe.dk/vamas-twa-20/). A plenary talk and other presentations were given at ECRS-5, Holland in September 1999.

5. Future Projects

None at present

6. Future Meetings

Further presentations will be made at ICRS-6 which is to be held in Oxford, UK from 10-12 July 2000. The next meeting of VAMAS TWA 20 is scheduled for 13-14 July 2000 at ISIS, Abindon, UK.

TWA 21 - Mechanical Properties of Hardmetals

Chair: Dr Bryan Roebuck
CMMT, NPL, Teddington, Middlesex TW11 0LW, UK

Annual Report - April 2000

Activity Review (1999-2000)

Chair and Vice-chair remain unchanged - Dr Roebuck, NPL, UK and Dr Oakes, Teledyne Advanced Materials, USA.

The completed project on bend testing resulted in modifications to a documentary testing standard, ISO 3327, Bend Strength Tests for Hardmetals. The revised standard is now out for voting as a Draft International Standard (DIS) with ISO member states, having been reviewed and approved at an ISO Committee meeting in 1999. The TWA 21 group includes direct participants and correspondents.

A new project has been formulated on toughness testing. Following wide dissemination in articles for appropriate technical press articles a core group that includes at least 10 industrial laboratories from 8 countries has agreed to participate in a test schedule. The plan is attached. The first materials for testing have just been received at NPL, so the timetable given in Appendix A has been delayed by about 3-4 months.

TWA meetings were held at international conferences on Powder Metallurgy in the USA and in Italy in 1999. The next opportunity for a meeting will be at the Powder Metallurgy Seminar in Reutte, Austria at the beginning of 2001. Local dissemination groups include trade associations in the UK, Germany and USA. These groups generally meet several times every year and progress reports are tabled by TWA member participants.

Possible Future Project

- Comparison and evaluation of Vickers and Rockwell Hardness Test

  Rationale:- confusion amongst hardmetal customers in Europe and North America where specifications may require different methods for hardness measurements.

VAMAS Project - TWA21
Toughness Tests for Hardmetals

Outline Plan

Objective

To evaluate different toughness tests for hardmetals, including the Palmqvist indentation test.

Participation

Material supply and testing:

National Physical Laboratory	UK	Sandvik Hard Materials	UK
Dymet Alloys	UK	Marshalls	UK
Teledyne Advanced Materials	USA	Kennametal	USA
Plansee Tizit	Austria	BAM	Germany
Harditalia	Italy	Baildonit	Poland
Boart Longyear	Eire/Germany	United Hardmetals	Germany
Hughes Christensen	USA	Hilti	Liechtenstein

Collaboration/Dissemination Groups:

British Hardmetal Association Research Group
USA Cemented Carbides Association
Japan Cemented Carbides Association
German National Carbide Association

Rationale

The interlaboratory exercise will generate underpinning technical information on well characterised materials that will allow good practice for toughness tests for hardmetals to be specified. It will especially provide a wider understanding of the benefits of short bar/rod chevron notched beam (CNB) tests vis a vis single edged precracked beams (SEPB) and provide guidance on the allowable range for useful Palmqvist tests.

Following circulation of an analytical report of the exercise, an ISO Technology Trends Assessment document will be produced as a first step in recommending appropriate suitable test methods that have the wide acceptance of industry.

Plan

A subset of the participating organisations will supply materials for tests in the form of rectangular bars or rods, dependent on test method to be evaluated. Further subsets of these will be used for Palmqvist tests.

NPL will coordinate materials supply and preparation of Palmqvist test pieces. Participating organisations will be sent two sets of samples for Palmqvist tests, one already indented and one with as-ground surfaces. Participants will polish, indent and measure toughness by their own procedures on this second set as well as measuring the first set. Table 1 gives the material supply and testing matrix for each organisation. Table 2 gives the material types. Table 3 gives a breakdown of the in-house test schedule. In-house tests will be performed by appropriate organisations on three sets of material:

- Their own grades
- Two additional grades tested by everyone; to be selected
- One further grade, randomly selected.

Table 1

Materials Supply and Testing Matrix

Organisation	Material Supply Y/N	Palmqvist Tests Y/N	In-house Tests Y/N
NPL	N	Y	Y
BAM	N	N	Y
Dymet	Y(3)	Y	N
Sandvik Hard Materials	Y(2)	Y	Y
Kennametal	Y(3)	Y	Y
Baildonit	Y(2)	N	N
United Hardmetals	Y(2)	Y	Y
Hilti	N	Y	N
Harditalia	Y(3)	Y	N
Boart Longyear	Y(2)	Y	Y
Teledyne	Y(2)	Y	Y
Plansee	Y(2)	Y	N
Marshalls	N	Y	Y
Hughes Christensen	N	N	Y

Table 2

Material Types

Organisation	Code	Material	Organisation	Code	Materials
Sandvik HM	HM1 HM2	Ultrafine Fine/alternative binder	Baildonit	B1 B2	6% Co, submicrometre 10% Co, submicrometre
Teledyne	T1 T2	Medium, low cubic, 10-11%Co As above + Ruthenium	Harditalia	H1 H2 H3	Low Co Med Co High Co
Plansee	P1 P2	Submicrometre hardmetal Cermet (Palmqvist only)	Kennametal	K1 K2 K3	6% Co, small gs, low cubics 8½%Co, med gs, high cubics 9½%Co, large gs, no cubics
Dymet	D1 D2 D3	5% Co; fine/medium; 9% cubics 7½% Co; medium; 20% cubics 6% Co; fine/medium	United Hardmetals	U1 U2	10% Co, 0.8 μm gs 3½% Co, 0.8 μm gs
Boart Longyear	BL1 BL2	7½% Co; medium; 20% cubics High/medium Co; coarse WC			

Timetable

- Communicate outline plan to participants July 99
- Produce materials for test May 00
- Coordinate and supply testpieces (NPL) Aug 00
- Complete test schedule Mar 01
- Preliminary analysis for discussion June 01
- Final Report Oct 01
- Recommendations for Good Testing Practice Feb 02

Test Schedule

All materials will be tested for Palmqvist toughness, but only subsets will be tested by in-house methods. Those organisations supplying materials and able to test in-house will do so on their own hardmetals. Other organisations able to do in-house tests will each do so on 6 materials, with some overlap as indicated in Table 3.

Table 3

In-house Tests

Organisation	Material Supply Y/N	In-house Tests on Own Materials Y/N	In-house Tests on Alternative Supply - Codes to be agreed*
NPL	N	N	Y
BAM	N	N	Y
Dymet	Y	N	N
Boart Longyear	Y	Y	Y
Teledyne	Y	Y	Y
Baildonit	Y	N	N
Plansee	Y	N	N
Harditalia	Y	N	N
Kennametal	Y	Y	Y
Hilti	N	N	N
Sandvik HM	Y	Y	Y
United Hardmetals	Y	Y	Y
Marshalls	N	N	Y

* The schedule for in-house tests will be circulated with an accompanying letter to individual participants.

The test schedule requires some organisations to prepare materials for collaborating companies to test. The dimensional requirements for appropriate in-house tests are given in Table 4.

Table 4

In-house Test Piece Requirements

Organisation	Test**	Dimensions, mm*
NPL	SEB	2 x 5 x 35 (min)
BAM	SEB	3 x 4 x 45
Boart Longyear	Terratek short rod	Nominal 10 ϕ x 15 long
United Hardmetal	SEB	6 x 10 x 20
Teledyne	Terratek short bar	Nominal 12 x 12 x 18
Kennametal	SEB	3 x 6 x 45
Sandvik HM	SEB	2 x 5 x 35
Marshalls	Terratek short rod	Nominal 12.7 ϕ x 19 long
Hughes Christensen	Terratek short bar	Nominal 12 x 12 x 18

* 3 test pieces/material grade
** SEB - Single edge beam, notched or precracked

VAMAS TWA 22

Mechanical Property Measurement of Thin Films and Coatings

Chairperson: Dr Stuart Saunders

National Physical Laboratory, UK

April 2000

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2.2 Project Leaders

Project 1: *Measurement of Hardness and Young's Modulus of Thin Coatings Using Depth Sensing Indentation Instruments*
Stuart Saunders, NPL.

Project 2: *Adhesion Of Thin Coatings*
Douglas Smith, NIST

3 OVERVIEW OF ACTIVITIES

Progress on analysis of data produced in the depth sensing indentation project (Project 1) has been made. This work is not straightforward due to lack of good calibration data that should have been provided by the participants. Nevertheless, it has been possible to agree on an approach to the data analysis that will yield useful information. The report in expected to be available in autumn 2000.

Considerable interest has been generated in the new project on adhesion (Project 2). Although the initial attempts to organize a round robin with TiN coated samples were unsuccessful due to the nature of the samples, this has encouraged a great deal of debate as to the best way forward. A new mailing list has been created of people known to TWA 22 to be interested in adhesion measurement problems. An email survey (Appendix, below) consisting of ten questions was sent out to several hundred people in early April 2000. About 40 people have already responded and expressed an interest in participating in the activity.

4 STATUS OF CURRENT PROJECTS

4.1 Measurement of Hardness and Young's Modulus of Thin Coatings Using Depth Sensing Indentation Instruments - *Project 1*

Project Leader: Stuart Saunders,
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Project Objectives

Development of methodologies for the determination of the mechanical properties of a coating *in situ,* and

Validation of the method by carrying out an international round-robin.

Progress

Progress in the analysis of the indentation loading-unloading curves has been made using the NPL spline fitting methodology. This provides a consistent method to determine the unloading slope required for calculation of sample stiffness (Young's modulus). The method has also been successfully applied to the analysis of data produced by an AFM for the determination of the indenter area function. Analysis of data provided by the participants using this methodology is now in progress. Unfortunately, not all participants provided sufficient calibration data, so a complete analysis will not be possible. An outline for the final report on this project has been agreed upon, and the report is expected to be completed by the autumn.

Standardization

Information obtained in this project has been fed into standards now being written for depth sensing indentation in 2 ISO TCs and 2 CEN TCs.

Dissemination

A number of talks and meetings have been given in the UK and USA relating to the work carried out in this TWA: Some are listed below:

- Invited speaker to the 2nd European Nanotest Indentation Workshop (Birmingham) (July '99)
- Surface Technology launch meeting (NPL, July 99)
- Invited speaker at SURCON99 (Birmingham, Oct. 99)
- Coatings workshop at NIST (August 99)
- Colloquium at IBM Yorktown heights (Dec. 99)
- Colloquium at Naval Research Laboratory Washington DC (Dec. 99)
- ICMCTF 2000 in San Diego

4.2 Adhesion of Thin Coatings - *Project 2*

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Project Objectives

To evaluate and compare several test methods for adhesion of thin coatings (<10 μm), including bend testing and indentation, through round robin testing, with the long-term goal of developing a simple, quantitative engineering test for coating adhesion.

Project Progress

Samples were supplied by NRIM in Japan for use in a VAMAS study of test methods of adhesion. The plan for the work was to produce two coating types that were well bonded and less well bonded to the substrate (AISI 316 stainless steel). The coatings were then to be evaluated by partners in an international round-robin where each partner performed a tensile test specified by the VAMAS TWA and any other tests that would give quantitative information about the interface. Before issuing the samples to partners, it was decided to carry out a "mini-round-robin" to ensure the proposed test method was applicable to the coated samples. This report summarizes the results of this initial phase of the project.

Sample Production

TiN samples were produced by reactive ion plating using 4 Ti targets and high purity N_2; the variation in adhesion was obtained by altering the thickness of the Ti interlayer that is usually employed to promote adhesion. The substrate temperature was maintained at 500±20 °C. The deposition rate was 0.05 μm/min. For good adhesion the Ti interlayer was 0.1 μm thick, while for poor adhesion the Ti layer was too thin to be measured. Coating thickness was determined using the Calotester and found to be 3.2±0.3 μm for both coating types. 10 x 100 x 3 mm, bars coated on both sides are available for each coating type.

Four-Point Bend Tests

The theory behind this test method relies upon the fact that during tensile straining cracks are generated and ultimately the crack spacing obtains a minimum value when spallation occurs. The crack spacing at the point of spallation can the be related to the interfacial shear strength (Figure 1). Initial experiments indicated that the TiN films did not spall during the bending experiment as required by theory (Figures 2 and 3), so that quantitative measurement of interfacial shear strength could not be carried out. The four-point bend test configuration did not allow the samples to be strained to greater than about 10%. In Figure 3 it should be noted that there is evidence of a secondary crack pattern at approximately 45 degrees to the primary cracks.

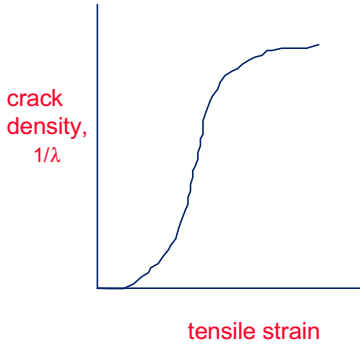

Figure 1 Schematic diagram of crack density versus applied tensile strain and the relationship of interfacial shear strength to measurable coating properties

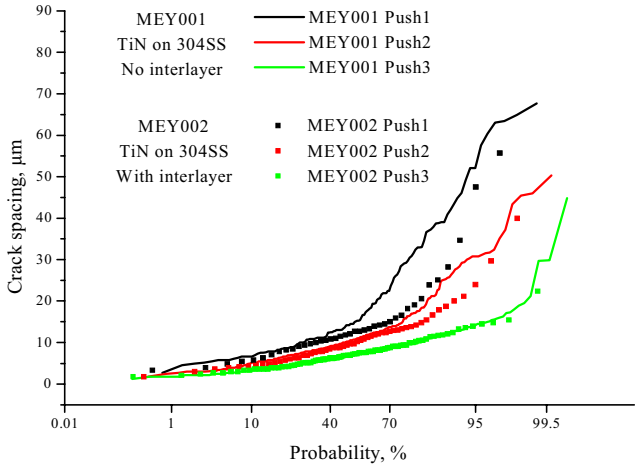

Figure 2 TiN coating cracking during four-point bend test to about 10% strain (push3)

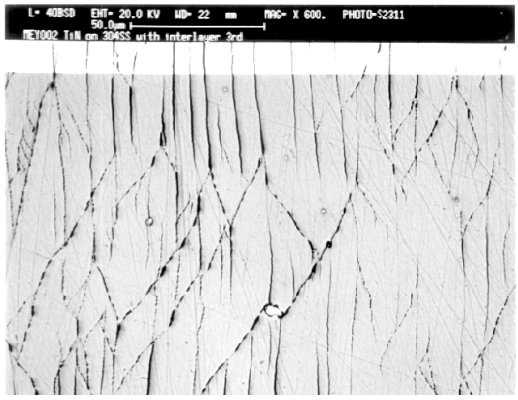

Figure 3 Backscattered electron image of TiN strained to about 10%

As a result of the relatively strong adhesion of all of the TiN coatings prepared, including those with “poor” adhesion, the project is being redesigned. A survey was sent out on April 4, 2000, to approximately 200 people in order to gauge interest in the project and to help the organizer determine which coating systems and which adhesion tests are of greatest interest to the thin film community. The survey and its introductory letter are provided in the Appendix. To date, forty positive responses have been received, with suggestions for coating systems and test to be studied. Preliminary results to the survey questions were discussed at the VAMAS 22 evening meeting on April 10 at ICMCTF00 conference in San Diego. Because of the very wide range of both coating-substrate systems and adhesion tests, the most likely course of action will be to perform several separate round-robins with several different coating systems.

Standardization

New work items on adhesion measurement have been agreed by CEN TC 184 WG5, and the progress to be made in this project will directly input into this activity.

Dissemination

There has been no formal dissemination of results to date.

5 PROPOSED/FUTURE PROJECTS

There are at the moment no additional formal proposals for new projects. It is generally agreed that a project on residual stress measurement and other non-destructive test methods for thin film mechanical properties (elastic properties by surface acoustic wave techniques, for example) should be proposed, but only when Project 1 is complete. Dr. Uwe Beck, of BAM in Berlin, is preparing a survey on these methods, and is expected to submit a formal proposal when Project 1 is complete.

6 FUTURE MEETINGS

- The ICMCTF 2001 will be used again as the main meeting for this TWA.
- The PSE meeting in Germany provides a further opportunity for discussion and publicizing VAMAS.
- Other opportunities are expected to arise during the course of the year.

Appendix: Adhesion (Project 2) Email Survey

Text of Introductory Letter:

Dear Colleague:

As many of you may know, the organization known as VAMAS (Versailles Project on Advanced Materials and Standards) has for several years now had a Technical Working Group (TWA 22) that deals with international efforts to develop standard test methods for measuring the mechanical properties of thin films and coatings. The first project in TWA 22, begun about three years ago and still underway, involves the measurement of film hardness and elastic modulus using depth-sensing, or instrumented, indentation. A large round-robin test was organized and carried out on aluminum and aluminum oxide films, with over 40 laboratories participating. Results are still being analyzed and a report is being prepared. The project was organized and executed by Stuart Saunders and Nigel Jennett at NPL, Doug Smith at NIST, and Toyonobu Yoshida at the University of Tokyo, with additional help in specimen preparation and analysis from Takahito Ohmura at NRIM.

Two years ago, a second project was approved in TWA 22 to evaluate and develop adhesion test methods for thin films and coatings, again primarily through international round-robin tests on common sets of specimens. This project was begun in response to what is perceived as a widespread need for quantitative, standardized adhesion tests for thin films and coatings.

A considerable amount of work has been done over the last 12 months in preparation for the intercomparison on adhesion test methods. TiN coatings deposited on stainless steel were produced by NRIM as 10 x 100 x 3 mm bars. The aim was to produce two types of coating with different levels of adhesion by varying the thickness of the Ti interlayer between the substrate and the TiN coating. The samples were designed to encourage as many participants as possible to undertake four-bend testing and determine the crack spacing at the onset of spallation and thereby derive the interfacial shear strength. In addition, it was intended that participants use any other adhesion test that may be appropriate with these samples. Preparatory work with these samples to refine protocols for the intercomparison found that it was not possible to induce spallation of the coating during the four point bend test. Other tests tried were scratch testing and indentation. NRIM reported that the two types of coating could be distinguished using the scratch tests but this could not be reproduced at NPL. Indentation testing also yielded negative results. It was therefore concluded that these TiN coatings would be unsuitable for a round robin. The TWA 22 Chairmen are therefore looking for alternative coatings. If you have reproducible coatings and would like the idea of (free) test results obtained by different adhesion tests and by different laboratories, please make contact.

The challenge now is, therefore, to select a new set of film/substrate systems, with tests appropriate for those systems that yield quantitative data, from the array of adhesion and adhesion measurement problems faced by industry.

Another immediate goal of TWA 22 is to determine where the greatest needs are in adhesion testing, whether it be in developing new tests or in providing better modeling, quantification or standardization of existing tests.

Towards this end, I have at the end of this email a short list of questions regarding any adhesion testing you may currently be involved in. I would greatly appreciate it if you could look over this informal survey and send back a quick response to

douglas.smith@nist.gov.

If I've missed my mark by putting you on this email list, and you don't want to hear any more about the project, just let me know, and I'll remove your name. If you're not interested, but know someone else in your organization who would be, please feel free to forward this survey to them.

The next meeting of the VAMAS TWA 22 group will be Monday evening, April 10, at the ICMCTF00 meeting in San Diego. We will be discussing both the indentation and adhesion projects. If you're going to be at that conference, and are interested in either topic, please join us. Check the message board when you arrive for details of the time and room.

If you plan to answer the questions below, please do so as soon as possible. I'm hoping to have some responses back before the San Diego meeting.

Thanks very much for looking all this over. The questions follow below.

Sincerely yours,

Douglas Smith, VAMAS TWA 22 Project 2 Organizer

Ceramics Division
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Gaithersburg, MD 20899 USA
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Phone: 301-975-5768
Fax: 301-975-5334

Informal Survey Questions:

1. Do you currently work with adhesion testing, or with film or coating systems where adhesion measurement or failure is an issue?
[Yes/No]:.....

If 'No', you're done, except perhaps to forward the survey to any colleagues to whom it would be more relevant. Thanks.

2. How adequate do you feel current thin film or coating adhesion measurement techniques within your field of research are?
[1= inadequate; 3= barely adequate; 5= completely adequate]
a) qualitatively? .....
b) quantitatively?.....

3. If adequate tests are available within your organization, please specify the tests:

4. Would you like to see the tests be nationally or internationally standardized?
[please check one -]
Nationally: .....; Internationally:.....; Neither:.....;

5. Do you require quantitative results from an adhesion test?
[Y/N]: .....

6. How sufficient is qualitative ranking or comparison to an in-house reference system?
[1= almost never; 3= sometimes; 5= nearly always]: .....

7. With what basic types of coating/film systems do you work:
[check those that apply]

Brittle coatings on Ductile substrates;.....,
Brittle coatings on brittle substrates;.....

Ductile coatings on brittle substrates;.....
Ductile coatings on Ductile substrates;.....

Polymer coatings or adhesive interlayers;.....,

Multilayers: all ductile:.....; all brittle;.....; mixed components;.....

Others (please specify)

8. What adhesion tests do you currently use (be specific if possible, otherwise generic test types will do)? If any of these are national or international test methods, please identify the tests (e.g. N= National, I= International).

9. If a national measurement laboratory like NIST in the US or NPL in the UK were to work on developing, quantifying or standardizing a small number of adhesion test methods, what kinds of tests or film/substrate systems do you feel would be of the most value to you? [Please specify]:

10. If an international round robin test were to be conducted, either to compare several test methods or to work toward quantifying specific test methods, would you be willing to participate, with the assumption that the test is one that you can perform?
[Yes/No]: .....

End of Survey. Thank you for your comments.

ANNUAL REPORT OF TWA 23

THERMAL PROPERTIES OF THIN FILMS

Chairperson: Albert Feldman, NIST
March 13, 2000

TWA 23 Project Leaders and Officers:

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Brief Overview

Measurements for a round robin to measure the thermal conductivity of silica films on silicon wafer substrates have been completed. The analysis of the round robin results is complete and a draft of the report describing the round robin is nearing completion.

Status of Current Projects

Project number 1

Title: A round robin to measure the thermal conductivity of silicon dioxide films on silicon

Project Leader and Affiliation

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Project Objectives

To evaluate measurement methods for determining thermal conductivity of thin ceramic films.

Progress Since Last Report

The round robin to measure the thermal conductivity of silica films on silicon substrates has been completed. Films of silicon dioxide on silicon with nominal film thicknesses of 50 nm, 100 nm 200 nm and 500 nm were chosen. 21 laboratories were sent specimens. Each laboratory chose its own measurement methods. For various reasons, 10 laboratories were unable to successfully perform the measurements. Eleven laboratories returned results that

were reportable. The analysis of the round robin results is complete and a draft of the report describing the round robin is nearing completion.

Standardization Activities

Through one of our participants, Dr. Apostolescu, we are maintaining contact with ASTM Committee E37 on Thermal Measurements.

Talks/Reports/Publications Based on Project Results

An oral presentation describing the results of the thin film thermal conductivity round robin will be presented at a special session on Thin Film Thermo-Physical Properties to be held at the 14th Symposium on Thermophysical Properties being held June 25-30, 2000 in Boulder CO.

Proposed/Future Project

No new projects are planned. We have examined the question of holding a round robin to measure the thermal conductivity of thermal barrier coatings but a survey of companies in the U.S. that produce these coatings suggests insufficient industrial interest.

List of Future National/Regional/International meetings relevant to TWA activities, including meetings where TWA results are presented.

A special session on Thin Film Thermo-Physical Properties to be held at the 14th Symposium on Thermophysical Properties being held June 25-30, 2000 in Boulder CO.

TWA 24

Performance Related Properties for Electroceramics

Chairperson: M. G. Cain, NPL, UK

Annual Report: April 18, 2000

TWA #24 Officers:

Chairman:
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Co-chairman:
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DMX/LC (Laboratoire de Ceramique)
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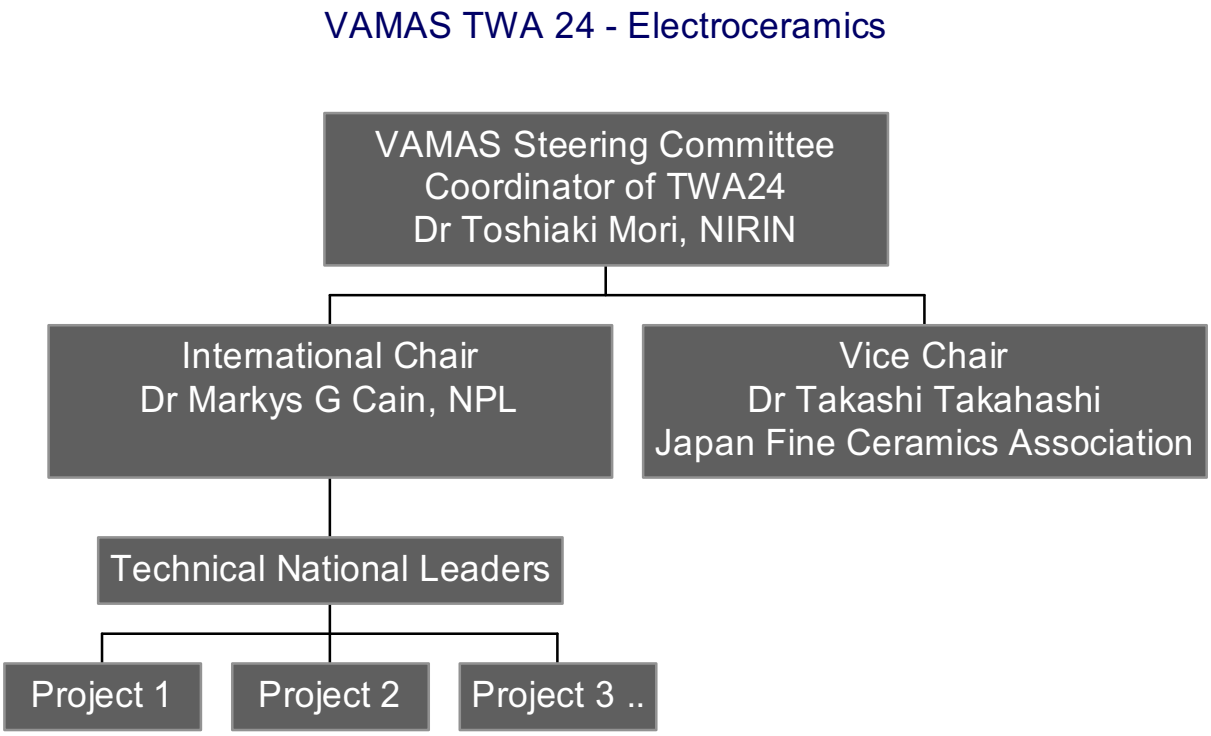
General Overview

Electroceramics such as piezoelectric and electrostrictive materials are materials which have the capability of converting electrical energy into mechanical energy (or vice versa). The technological importance of these materials is increasing, with widespread applications in actuator and sensor applications. Although there has been some standardisation activity through bodies such as the IEEE and more recently through CENELEC in the development of new standards, many of the most important properties that are required for these materials when used as sensors and actuators remain without internationally recognised test methods. Measurements where work is required include direct and converse piezoelectric coefficient measurement, high stress dielectric property measurement, the measurement of strain at high stresses, and the measurement of degradation of materials performance under repeated electrical and mechanical loading.



The TWA is working to enlist to co-operation of measurement laboratories that are interested in evaluating methods for determining performance-related properties of electroceramics. Initially it is expected that efforts will focus of development of procedures for the following project:

Project 1	Measurement of piezoelectric coefficient of materials of differing geometries and configurations, including assessment of direct and converse coefficients

Materials: Piezoelectric materials

Status of VAMAS 24

The concept of a new TWA in electroceramics pre-standardisation was taken in January 1999. An email based questionnaire was written and sent to major electroceramic users and producers around the world, Appendix 1. There was overwhelming support for the setting up of this TWA. A joint meeting with TWA 3 chairman, George Quinn, in Brighton 1999, concluded that a separate (rather than joint ceramics) TWA should be arranged. This was approved at the Steering Committee meeting in 1999.

The projects that were supported from the email questionnaire were further ratified by the Japanese response to the formation of the new TWA. In this response (from Toshiaki Mori - NIRIN), the Japanese industrial researchers position was clarified and projects were described in enough detail for our first project initiation form to be completed. This project number 1, is the Measurement of the piezoelectric coefficient of Materials, and its project initiation form has been sent to Dr Mori, and also the National Coordinators for expressions of participation and interest to be gained and steering committee approval.

The probable UK industrial involvement would include: Morgan Electroceramics, Queensgate Instruments, Solartron, ACL Ltd, GEC Marconi Caswell, and others. Academic involvement would include some of our major functional materials laboratories at Cranfield and Manchester for example. This support still needs final approval. International support and participation in this project would be based on the initial database of supporting organisations and of course that gained by our National Leaders. The Japanese National Leader, Dr Takahashi, will manage Project 1.

A new National Leader from Latvia, a Dr Andris Sternberg, Director of the Ferroelectric Physics Department at University of Latvia, has been nominated by Dr Cain and we await the Steering Committee's approval.

The global awareness and impact of VAMAS has been raised at a recent Office of Naval Research Review (April 2000 at Penn State University- Project Manager Dr Wallace Smith). Much interest was demonstrated and potentially new project ideas and participation. This kind of activity will form a main dissemination thread for TWA 24.

PROPOSED FUTURE PROJECTS

The following projects have been proposed and are at an early planning/discussion phase.

Project 2	measurement of piezoelectric strain at high electrical/mechanical stress
Project 3	measurement of piezoelectric and dielectric properties at high stress
Project 4	measurement of electrical and mechanical fatigue of piezoelectric ceramics materials
Project 5	properties of electrical conductive, optical transparent thin films
Project 6	thermal effects on performance
Project 7	the dielectric, elastic and piezoelectric properties (matrix elements) need to be measured as complex coefficients so as take account of the electrical, mechanical and piezoelectric losses in the material. The complex coefficients should and can be measured as a function of frequency.
Project 8	The temperature dependence of the complex coefficients is important in some applications (such as high power and space applications).
Project 9	hydrostatic property measurements to about 14 MPa are important for underwater applications.
Project 10	the meaning of fatigue / aging / degradation were clarified, particularly the distinction between reversible and irreversible mechanical or electrical damage
Project 11	In project 1 it would be interesting to introduce measurements of piezoelectric coefficient of thin and thick films on substrates.

OTHER

Interactions with Technical Working Area #3, Ceramics for Structural Applications
Mr. Quinn has been in contact with Dr. Cain regarding the new TWA. The TWA 3 national leaders agreed that a new TWA was appropriate. Mr. Quinn met with Dr. Cain in London in June 1999 a few days before the TWA 3 meeting and they agreed agree to coordinate their respective TWA's. Dr. Cain came to the Brighton TWA 3 meeting and spoke to the members about his plans. Dr. Mizuno then

proposed four possible projects for TWA 24. Three of these projects matched those that originated from the email based questionnaire, organised by Dr Cain.

The VAMAS TWA 24 web is accessible from the following home page:

http://www.vamas.org/

The site includes a single page summary of the TWA 24 projects as well as general descriptions of TWA 24 and VAMAS.

The TWA 24 home pages are likely to be created at the Functional Materials Group Website at NPL.

Dr Markys G Cain
18 April 2000
National Physical Laboratory, UK.

VAMAS TWA 25

CREEP/FATIGUE CRACK GROWTH IN COMPONENTS

CHAIRMAN: Dr. Kamran Nikbin
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London, SW7 2BX, UK

10th April 2000

Co-Chairman: Professor T. Yokobori
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2. Project Leaders

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3. Overview

VAMAS TWA 25 began in July 1999. The overall aims of this workgroup is as follows

- Establish accurate and reliable procedures for assessing creep/fatigue crack growth at elevated temperatures in components which contain defects.
- Determine procedures for analysing the test data using fracture mechanics concepts.
- Validate results against measurements on standard laboratory specimens using the ASTM E1457-98 testing procedure and the new BS 7910 assessment procedure
- Outline recommendations in procedures for dealing with component creep/fatigue crack growth testing and analysis

Currently TWA 25 is concerned with collecting a data base and information regarding participant's activities and experience in the field of creep and creep/fatigue crack growth in components.

4. Status of current projects

Since the start of the project two meetings have been held. The kickoff meeting was in London in June 1999 when 15 people attended. The aims set out above were discussed and agreed upon. Eight oral presentations were made and a questionnaire was distributed to collect information from partners who have interest in the project. The second meeting was held in Tokyo in Nov. 1999 where the results of over 20 question sheets that were returned, were discussed. There were oral presentations by a number of participants. It was agreed at the meeting, on the basis of information collected from colleagues, that a second more detailed questionnaire will be prepared to collect information regarding matters concerned with component testing. This has now been sent to those participating in the project. The details of the minutes, future meetings and additional information about participating information is held on the **VAMAS TWA 25** website at http://mesm.org

It has also been agreed by partners that the main method of communication will via e-mail. Therefore an e-mail list of all participants is provided at the bottom of this report.

A local meeting was held with the Co-Chairman and the Japanese VAMAS committee in April 2000 to discuss strategies for collaboration with Japanese Industry. Finally a 3rd open meeting has been arranged for October 3rd 2000 at MPA Stuttgart. The details are placed on the web site.

Ongoing work

Title	Completion date	Output
collect shared information on participants interest	Nov-99 to Nov 00	Data-base
collect detailed information on creep and creep/fatigue component testing and analysis	April 00-October 00	Data-Base Analysis

5. Other Planned Output in the Next 3 Years

The overall plan for the future is outlines as follows

- gather together experts from industry and research institutes in order to identify their specific needs with respect to feature component testing.
- produce a data-base of available feature component tests and interpreted data.
- identify acceptable feature components and best practice for undertaking tests.
- produce recommendations for analysis of component data.
- establish reliable methods for prediction of component behaviour.

Meetings

1) Kickoff meeting	London, June 1999	15 participated
2) 2nd Meeting	Tokyo, Nov. 1999	20 participated
3) meeting with Co-Chairman	Nagoya, April 2000	5
3) 3rd meeting	Germany, Oct. 2000	(to be held)

TWA ANNUAL REPORT

TWA 26

Full field optical stress and strain measurement

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Monday, 17th April 2000

TWA Officers

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TWA Project leaders

Project leaders to be determined at the first meeting to be held at the SEM conference in Orlando, Florida, USA on 5-8 June 2000. A session devoted to the activities of VAMAS TWA 26 is being held on Thu 8th June from 09:00 to 10:30.

Summary

The first meeting of the TWA officers and participants will be held on Thursday 8th June 2000 at the SEM conference in Orlando Florida, USA. The 1.5 hour session will comprise the following activities

AGENDA

1. Introductions (Eann Patterson , co-chair)
2. Rationale for Standards on Optical methods for Stress and Strain Measurement. (Richard Burguete, co-chair)
3. Presentations:
    a. North American Perspective (Michael Sutton)
    b. European Perspective (Michel Honlet)
4. VAMAS TWA 26:
    a. Participants
    b. Modus Operandi
    c. Projects
5. Date of Next Meeting
6. Any other business

This will be the first opportunity to determine how to group the common areas of interest and distribute projects accordingly.

A web page is being prepared to present the remit and activities. The web address *www.twa26.org* has been purchased and a site will be set up on the University of Sheffield server. This web page is proposed to be ready and published by the end of May 2000.

A list of invited participants is given below and their respective areas of expertise/activity have been detailed. The participants listed are those who have responded to the initial invitation to join in the TWA.

Current project status

Projects have not yet been agreed but the following table shows the matrix of activities which will be grouped into projects at the first TWA meeting in June.

MATRIX OF OPTICAL TECHNIQUES

	TECHNIQUES	Laser techniques					Moiré			Photoelasticity		Thermoelasticity
	SUB-TECHNIQUES	Caustics	Speckle Inf.	Shearography	Holography	ESPI	In-plane geom.	Out-of-plane geom.	Interferometry	Reflection	Transmission	
FIELDS	SUB-FIELDS											
Light sources	Spectral distribution											
	Temporal stability											
	Poly/mono-chromatic											
	Lasers											
Sensors	Detector type											
	Chip size											
	Pixel shape											
	Pixel response char.											
	Digital/Analogue											
Optical components												
Digitiser cards	Resolution											
	Speed											
	Noise											
	Data Compression											
Physical ref. mater.	Polarisers & ¼ waveplates											
	Gratings											
	Coatings											
Calibration materials												

MATRIX OF OPTICAL TECHNIQUES (CONT)

	TECHNIQUES	Laser techniques					Moiré			Photoelasticity		Thermoelasticity
	SUB-TECHNIQUES	Caustics	Speckle Inf.	Shearography	Holography	ESPI	In-plane geom.	Out-of-plane geom.	Interferometry	Reflection	Transmission	
FIELDS	SUB-FIELDS											
Virtual ref. maters.	Simulated data											
	Synthesised fringe patterns											
	Synthesis of noise											
Output standards	Image data format											
	Numerical data format											
	Processed data format											
Operational procedures	Specimen preparation											
	Application of surface media											
	Loading: range, strain rate etc											
	Ambient conditions											
	Specimen alignment											
Correlation methods												

Project number and title
N/A

Project leader and affiliation
N/A

Project participants
N/A

Project objectives
N/A

Progress (since last report)
As shown in the Gantt chart below in the activities 1-3 have been carried out. The activities 4,5 and 6 will be rolled up in to Milestone 7. The reporting structure and communication protocols (activities 8-10) have not yet been addressed and will be progressed during and after the first meeting.

Standardisation activities
N/A

Proposed/Future projects
As this is the first report and projects have not been decided yet there are no proposed or future projects.

Future meetings
SEM IX — International Congress & Exposition on Experimental Mechanics
5-8 June 2000, Radisson Hotel Universal Orlando, Orlando, Florida, USA.
For further information see www.sem.org.

Fracture Mechanics 2000 (Cape Town)
For further details contact Prof. Bob Tait, University of Cape Town
Increase international awareness of activities.

BSSM — Modern Practice in Stress and Vibration Analysis
5-7 September 2000 University of Nottingham, Nottingham, UK
For further details email a.a.becker@nottingham.ac.uk

Photomecanique 2001 at the Universite de Poitiers, France organised by GAMAC.
For details contact Prof. M. Cottron, Universite de Poitiers.

BSSM — Strain Measurement in the 21st Century
5-6 September 2001, Lancaster House Hotel, Lancaster, UK
For further details email bssmsec@moragmac.clara.net (by 1st October 2000)

International Conference on Experimental Mechanics 2002, to be held in Vienna. For further details contact: Professor Rossmanith.